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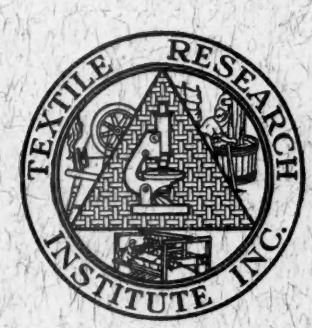
TEXTILE RESEARCH JOURNAL

VOLUME XVI May, 1946 *NUMBER 5*



PUBLICATION OF

TEXTILE RESEARCH INSTITUTE, INC.

AND

THE TEXTILE FOUNDATION

Editorial Office: 10 East 40th Street, New York 16, N. Y.

TEXTILE RESEARCH JOURNAL

VOL. XVI, No. 5 — May, 1946

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TEXTILE RESEARCH JOURNAL, published monthly at Prince and Lemon Streets, Lancaster, Pa., is devoted to the interest of research in textile and allied industries. It is the official publication of Textile Research Institute, Inc., and the Textile Foundation.

Manuscripts and communications should be sent to the editorial offices, 10 East 40th Street, New York 16, N. Y.

Subscription rates: General—$10.00 per annum; additional for postage, 50 cents to Canada, $1.00 to all other countries. Special—$5.00 per annum for United States and Canadian students, teachers, educational institutions, public libraries, governmental libraries and departments, plus 50 cents for postage to Canada. Single copies, $1.00 each.

Entered as second-class matter, April 27, 1945, at the postoffice at Lancaster, Pa., under the Act of August 24, 1912.

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TEXTILE RESEARCH JOURNAL

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Synthetic Fiber Developments in Germany, II

A Synthetic Fibers Team of the Technical Industrial Intelligence Corps, together with a team from the United Kingdom, in the summer of 1945 searched the synthetic fiber and film factories of Germany, Austria, Holland, and Italy for all information which might be of assistance to our industry.

The findings of the combined teams are contained in an official report, soon to be published under the above title. This report contains a 54-page summary which is intended for the reader who wishes to get only the general picture.*

The material in this article comprises the second half of the summary. The first part was published in the April issue, page 149.

The Acetate Process

1. *Acetic Anhydride*

The only process observed for making acetic anhydride was the one involving the dissociation of glacial acetic acid vapor to give ketene, followed by absorption in glacial acetic to yield acetic anhydride. The I. G. Farbenindustrie at Dormagen and Deutsche Rhodiaseta at Freiburg held licenses under the basic Wacker patents and, as could be expected, the general procedures were the same.

2. *Cellulose Acetate Flake*

The manufacture of cellulose acetate for filament yarns, staple fiber, and films does not seem to have been expanded very much during or just prior to the war.

The lack of cotton linters during the war period has doubtless hampered development, and most of the samples of acetate made from beechwood pulp gave solutions in acetone which were hazy and poor in color.

Although the German technicians considered that the quality of the yarn and the staple made from such acetate was adequate, they expressed a great desire for the return to cotton linters which would give them greater fiber strength and clearer films. Among the factories visited by the teams there were interesting differences in plants and techniques.

The Rhodiaseta plant at Freiburg, which had a capacity of about 275,000 pounds per week, relied on a small proportion of sulfuric acid as catalyst and a high proportion of acetic acid as diluent. This has some weaknesses; copper acetylators, running without agitation, may not produce very uniform batches, and wooden vats are prone to contaminate the product. It is surprising that no preliminary pretreatment was attempted here, in view of the low proportion of catalyst.

Alexander Wacker of Burghausen, producing some 60,000 pounds of flake per week, acetylate with a large excess of zinc chloride which acts as part cata-

*PB Report 7416. To be published by Textile Research Institute.

lyst and part solvent. This method eliminates the sulfuric acid and the formation of sulfoacetates which cause poor heat stability. On the other hand, the method is expensive and involves an elaborate process for the recovery of zinc chloride, thus complicating the acetic acid recovery.

The plant is very compact in that most operations up to the washing stage are carried out in one vessel which is similar to the xanthator-dissolvers used in viscose plants. The process is rather antiquated and does not inspire confidence. The samples of cellulose acetate flake examined did not give films of very good color, but were considered to be quite adequate for staple fiber.

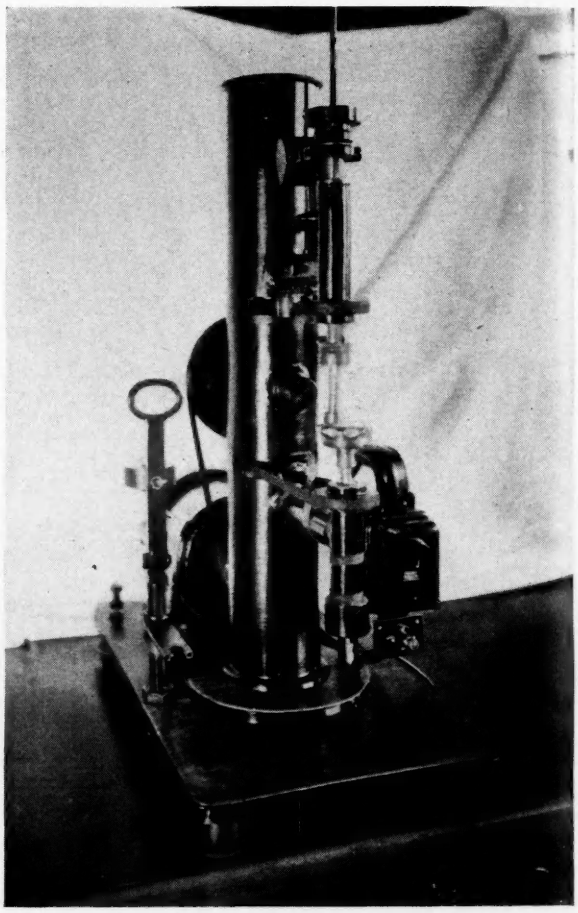
On the other hand, the I. G. Farben plant at Dormagen, producing up to 350,000 pounds per week of various grades, was considered to be very up-to-date and well planned. Here, the methylene chloride process is in operation; every precaution against corrosion seems to have been taken, and the product is good, considering the raw material.

The plant, which is massive, is constructed principally of aluminum and stainless steel. Every advantage is taken of compressed air and vacuums for moving the material through the production cycle. The continuous washing machine, coupled to a continuous centrifuge, is novel and worthy of imitation. The performance of the vacuum driers appears to be satisfactory.

In all the plants visited the designers have been bold enough to provide for large batches—the largest accommodations being at Dormagen. Despite the large size of the acetylators, elevated working temperatures, and the difficulty of temperature control, it was claimed that both the acetyl value and the viscosity could be controlled with fair accuracy. In any case, abnormally large batches could be absorbed in the ordinary course of business. The Burghausen and Dormagen plants provided a wide range of products to cover the needs of the yarn, lacquer, and plastics trades, while at Freiburg an elaborate blending system was in operation to even out the product.

From the points of view of labor costs per pound and output per unit of plant, the I. G. Farben factory at Dormagen holds a distinct advantage, and, given satisfactory cotton linters, we consider that their product would be very good indeed.

With slight differences, all plants used the same process for acetic acid recovery—*i.e.*, the extraction of weak acid liquor by a solvent in a stationary column, followed by rectification and recovery of solvent. At Burghausen, the extracting solvent was a mixture of 85 percent ethyl acetate and 15 percent benzene, while at Dormagen, owing to the presence of small quantities of methylene chloride in the acid, no benzene was considered to be necessary. At Freiburg, the older diethyl ether was used as the extracting medium.



This photograph of the latest "edge circular abrasion tester" appears on page 904 of the complete Report.

The plant at Freiburg is more intricate than the other factories. The equipment in all plants is very similar to that used in Great Britain and America, except that more use has been made of synthetic rubber and porcelain linings to reduce corrosion.

3. Spinning of Continuous Filament Yarn

The only plant inspected where continuous filament acetate yarn was being made was the Deutsche Rhodiaseta plant at Freiburg.

Dope is made up from 800-kg. batches of acetate roughly to approximate a 25-percent cellulose acetate solution containing 2 percent of water. The mixing

follows the usual procedure, as does the filtration which is done on the hot dope.

Spinning is carried out on double-sided machines, the spindles being on 6-inch centers. The spinning cells consist of metal tubes, heated by hot-water circulation. The cell carries the usual lantern. The spinning process is carried out by downward spinning, the air being introduced at the top of the cell and being removed near the bottom.

Individual gear pumps are fitted. The dope temperature is regulated independently of the air temperature. The yarn is wound up without twist on friction-driven bobbins at approximately 600 r.p.m.

The spinning machines are conventional in design, and the acetone recovery follows the normal practice of water absorption.

4. Staple Fiber Manufacture

Two plants which make acetate staple fiber were visited in Germany; they were the Deutsche Rhodiaseta plant at Freiburg and the Alexander Wacker plant at Burghausen.

In both plants standard machines for making continuous filaments are used. The filaments are collected at the end of the machine and conducted to a cutter. Deutsche Rhodiaseta used the Beria type of cutter, whereas Alexander Wacker employed a Snia cutter, using a current of water to give increased rigidity to the fiber.

In the Wacker factory 20-percent and 23-percent solutions are used for spinning. The air was preheated independently of the dope, the higher temperatures being used for the lower concentration. The dope itself is preheated by passing through a preheater on its way to the candle filter, which is suspended in the hot tube.

Square- and round-shaped cells of conventional design are used. Air is introduced concurrently with the yarn below the spinneret and withdrawn near the base of the tube. The fibers pass over eccentric rolls and are lubricated with water before cutting.

The whole of the product at Burghausen was being converted into wool-type fiber. This is manufactured by boiling the staple in an 8-meter-long bath with 1–2 percent soap. The staple is then dried, opened, and baled.

Bright staple has also been manufactured, but the crimp is put in mechanically. The staple for carding is treated with antistatic finish containing Igepon. The staple is said to have a tenacity of 1.46 grams per denier when dry, and 0.88–0.90 gram when wet.

Various types of dyed spun staple have been prepared in the following colors: black, blue, red, green, yellow, violet, and mixed shades from these basic colors.

The entire Rhodiaseta spinning plant was in good condition, but the Burghausen plant was dirty and the plant for production of wool-type crimped staple, together with the methods of handling, was out of date.

Only 1,343 tons of acetate staple fiber were produced in Germany in the year 1944 and the planned future capacity was only 4,680 tons. The lack of interest in this fiber is due to extensive requirements of corrosion-resistant materials and the high cost of acetic acid and anhydride which, along with the small scale of production, results in a selling price twofold or more of that of viscose staple. Actetate fibers are said by the German manufacturers to be of interest for their warmth, low moisture affinity, low density, and creaseproof properties. The deficiencies of acetate are high cost, difficulty in dyeing, low softening point, and difficulty during carbonizing when wool and acetate blends are reworked. In spite of these disadvantages it has been stated by impartial German experts that from the standpoint of quality acetate staple constitutes their best present chemical fiber for use in woolen-character garments. Nevertheless, the Nazi planners substantially prohibited its production.

5. Saponified Acetate Yarns

While some experimental work was carried out on acetate-type, high-tenacity yarns of the unsaponified and saponified types, there was no production of these yarns for military use as in Great Britain and the United States.

6. Cellulose and Cellulose Acetate Films

No advances have been made in the equipment for the preparation of the viscose and its subsequent casting into film; however, an effort is being made experimentally to adapt slurry steeping and continuous pressing, which are widely used in the production of viscose staple fiber.

Much of the research effort during the war period has been employed in developing suitable substitutes for the materials normally used for plasticization and moistureproofing.

Since the regular moistureproof cellophane was not entirely satisfactory for military purposes because of unsatisfactory performance at low temperatures and during sterilization, research has been intensified upon the development of superior anchoring and

coating materials. It is claimed that this has resulted in the production of a heat-sealing, moisture-proof cellophane which gives improved performance at *low* temperatures and also during sterilization.

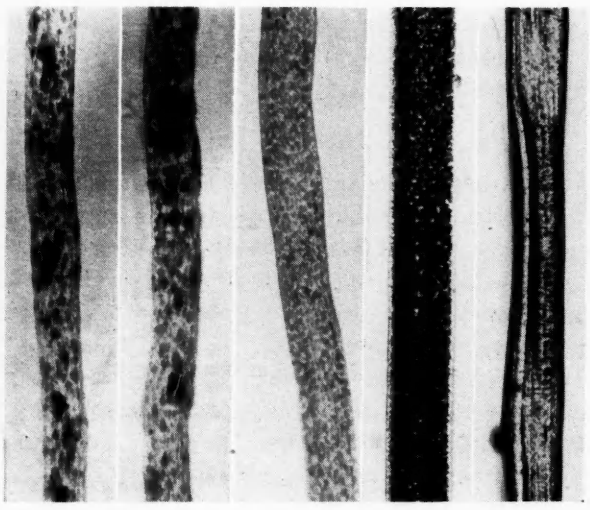
No comprehensive survey can be given on acetate films since only one plant (Burghausen) was seen. As elsewhere, the product has suffered from the poor quality of raw material used to make the acetate flake; samples from the current production exhibited bad color and poor physical properties. A booklet of prewar samples, however, showed a range which was equal to any produced in Britain or the United States.

Fully Synthetic Fibers

The German work on fully synthetic fibers has been divided among the polyamides (Perlons L and T), the polyurethanes (Perlon U), and, in the field of vinyl fibers, chlorinated polyvinyl chloride (PeCe fiber) which was first made about 1935. The polyurethanes are structurally allied to the polyamides, the recurring link being —NH.COO— in place of —NH.CO—. The use of vinylidene chloride as a starting material for fiber-forming polymers is not new, but the approach to this problem differed from the procedure used in the United States.

In short, the observers were unable to find evidence of the development of any radically new types of fully synthetic fibers.

In summarizing the status of wholly synthetic fibers in Germany, it can be stated that progress in the United States compares favorably with that of Germany.



Photographs of animalized fibers, from page 936 of the Report. The two specimens at the left are Cupralan (3.75 D.P.F.); the next two are Vistralan—C.W.W. (3.75 D.P.F.); the one on the right is Vistralan XT (3.75 D.P.F.).

The slower growth of the production of polyamide fibers of the nylon type may be accounted for by the fact that Germany had access to considerable quantities of silk for military purposes and that there was a critical shortage of construction materials.

Considerable progress has been made with the commercial production of after-chlorinated polyvinyl chloride, PeCe fibers, and determined efforts are being made to develop an industrial fiber which will have a higher softening point. The research being done on the copolymerization of polyvinylidene chloride with other polymers is expected to fortify the position of the German industry in this respect.

Both PeCe and Perlon L fibers played an important part in the German fiber economy during the war. Because of their functional properties, these fibers (alone or in combination with other fibers) were effective in conserving the dwindling supplies of cotton and wool.

Considerable research has been conducted on the manufacture of fibers from polyvinylidene chloride, polyethylene, and the polyurethanes. None of these has reached commercial production in the fiber form. The polyurethanes do not appear to be interesting candidates for wearing-apparel materials because of their rigidity, but because of their low water-absorption, resistance to chemicals, and high strength they may find use in industrial or technical fabrics.

It is apparent that the Germans have given considerable thought to the development of fibers designed to meet the various functional, chemical, and mechanical requirements in the industrial field.

Since the I. G. Farbenindustrie had made the principal developments in new, noncellulosic fibers, observations and inquiries were made to secure an overall picture of the progress in process development and applications of the new fibers in industry and in the military effort.

1. Perlon (Nylon)

The development of polyamides, known in Germany as Perlon (for fibers) and Igamide (for plastics), has proceeded along rather different lines than it has in the U.S.A. and Great Britain. The I. G. Farben plants have concentrated their main production on Perlon L, a polymer of aminocaprolactam, while Perlon T, a 66 polymer corresponding to nylon as produced in the U.S.A. and Great Britain, has so far received comparatively limited attention. The

expansion of Perlon was slow during the first years of the war, with the maximum scale of production of the order of 300 tons/month. More recently, strenuous efforts were being made to build up a total polyamide production of 1,200 tons/month with the provision or expansion of spinning plants at Berlin-Lichtenberg, Premnitz, and Landsberg, to be followed at a later date with a further expansion to 2,000 tons/month. The main production to date has been of continuous filament, but a very promising future was envisaged for Perlon staple.

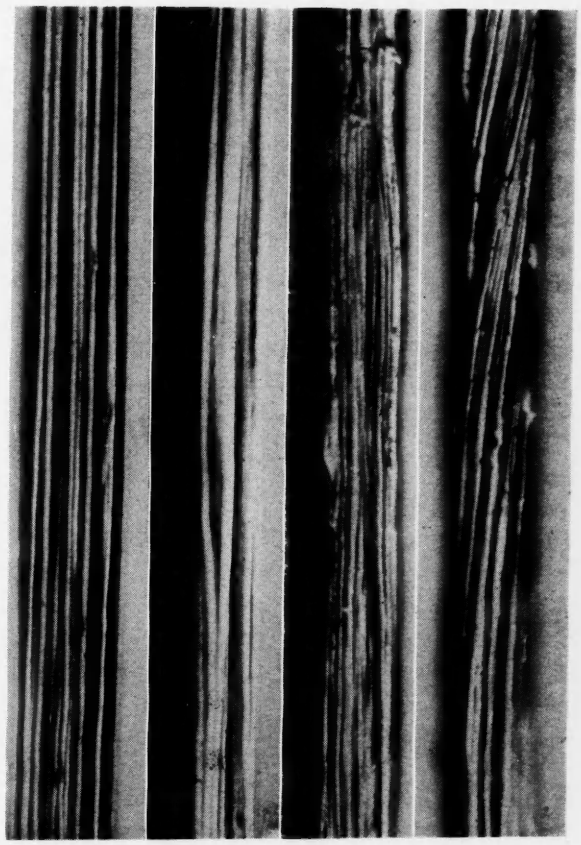
Some Perlon L was used in tire construction, but apparently without much success. Some was also used for parachute cloth, but as Germany had access to silk, little was used for this purpose.

The observation that Perlon staple is efficacious in prolonging the wearing qualities of fabrics when it is blended in small quantities with viscose staple, cotton, and wool emphasized its importance to the German national economy, and future plans were to be aimed at producing approximately three times as much staple as continuous filament. The benefits to be gained from staple admixtures of this kind were striking in the case of socks, in which the wear life was increased many times by the addition of 20–25 percent of Perlon fiber.

I. G. Farben has based its production of Perlons L and T on phenol obtained in the hydrogenation of brown coal. Owing to internal economics, benzene was a comparatively high-cost starting material, but phenol shortages during the war years had led to a study of benzene as the base product, and alternative processes had been worked out.

I. G. Farben claims that Perlon L is cheaper to produce than Perlon T, which is probably true under German conditions, and in the light of the Germans' knowledge of processes for making Perlon T intermediates. Plant capital costs were said to be in favor of Perlon L, while production could be expanded more rapidly. It was also claimed that Perlon L was easier to spin and process, but the degree of spinning efficiency obtained in practice did not justify these views, and it must be emphasized that I. G. Farben lacked sufficient experience in the manufacture and spinning of Perlon T to make a just comparison.

A fundamental disadvantage of Perlon L is the unfavorable monomer-polymer equilibrium, which gives rise to various processing difficulties and adds to the number of steps involved. For example, it was necessary in making continuous filament yarns to reduce the monomer content of polymer chips from approximately 10 percent to 1.5 percent in order to avoid sticking of the filaments. During the spinning of the molten polymer, 4–5 percent monomer again forms and must be washed out after spinning. Continuous-filament Perlon L compares unfavorably with Perlon T because of its lower softening point and inferior light-resistance.



These photographs are taken from page 937 of the Report. The two specimens at the left are regular viscose staple fibers and the two on the right are Cisalfa (animalized—casein addition to viscose).

Caprolactam used for Perlon L was produced by I. G. Farben at both Leuna and Ludwigshafen. Phenol was hydrogenated to cyclohexanol by well-established processes. Leuna employed an unconventional zinc-iron (10 percent) catalyst for oxidation to cyclohexanol, and claimed a 94 percent yield at 70 percent conversion.

The production of caprolactam from cyclohexanone by Beckmann transformation of cyclohexanone-oxime was on a scale of 200 tons/month at Leuna and 150 tons/month at Ludwigshafen. While the two processes are essentially similar chemically and in plant layout, it seemed to us that the procedure adopted

at Ludwigshafen was to be preferred (yield 85–92 percent).

Perlons L and T were produced on a 2-tons/batch scale at Ludwigshafen. A notable recent development is a simple and cheap process for continuous polymerization of caprolactam. Polymerization is conducted in a vertical 8-meter-long tube, at normal pressure; a strong aqueous solution of caprolactam is fed continuously to the top of the tube, and polymer is withdrawn continuously from the base. It was claimed that this process had been proved on the field scale, and that it had been the intention to utilize this at Premnitz for continuous production of 600 tons of Perlon L staple per month from caprolactam.

Adipic acid required for Perlon T was made both at Ludwigshafen (180 tons/month) and Leuna (120 tons/month) by nitric acid oxidation of cyclohexonal (85 percent yield). The processes differed slightly in operating details, but there were insufficient data to establish, unequivocally, which was the better.

Benzene had also been explored as a starting material for adipic acid, but none of the processes examined had been used for commercial production. The most important process was nitration and reduction to aniline, followed by concurrent vapor-phase, catalyzed reduction to cyclohexylamine, and hydrolysis to cyclohexanol. While the conversion of aniline to cyclohexanol involved some difficult by-product separations, a 98-percent yield was claimed. A 400-tons/month plant was destroyed by bombing before coming into operation. Another significant process was the air-oxidation of cyclohexane to a mixture of cyclohexanone with minor amounts of by-products, followed by nitric acid oxidation.

A more novel synthesis, still in the exploratory stage, was the reaction of tetrahydrofuran with carbon monoxide and water. Tetrahydrofuran was a comparatively low-cost material since it was produced on a very substantial scale at Ludwigshafen as an intermediate in butadiene manufacture from acetylene and formaldehyde.

The processes for producing hexamethylene-diamine from adipic acid show no outstanding features. An interesting new synthesis of hexamethylene-diamine also based upon acetylene and formaldehyde has been discovered at Leverkusen, which it was claimed would give, under German conditions, a lower-cost hexamethylene-diamine:

1,4-butine-diol, the primary product of interaction of acetylene and formaldehyde, produced at Ludwigshafen on a scale of 125 tons/day, is partially reduced to 1,4-butene-diol, which is then allowed to react with anhydrous hydrogen cyanide in the presence of a liquid cuprous-salt catalyst. The resultant 1,4-dicyanobutine-2 is reduced in two steps to hexamethylene-diamine. This process was still in the laboratory stage.

The development of polyamides and fibers from them has progressed slowly in Germany. Information developed indicates that the I. G. Farbenindustrie had a projected capacity of 31,680,000 pounds per year of *omega*-amino-caprolactam polymer and approximately 23,760,000 pounds per year of Perlon L fiber. Two-thirds of the fiber production was destined for Perlon L staple.

The decision to manufacture a considerable poundage of staple was greatly influenced by the national economy. It had been determined that the blending of small amounts of Perlon staple with viscose staple, cotton, and wool prolonged the wearing qualities, especially of hosiery. It was said that yarn containing 16 percent of Perlon staple blended with 84 percent Vistra viscose staple had the same durability as cotton yarn. The Germans were quite enthusiastic about the use of Perlon staple for increasing abrasion-resistance. It was predicted that the blending of 10–30 percent of Perlon staple with other synthetic fiber staples, cotton, wool, and silk will be extended to wearing apparel and certain industrial fabrics in the postwar period. The use of Perlon fibers with other fibers to decrease water-absorption and to promote quicker drying has also been tried. It was claimed that bathing suits made from yarns containing 20 percent Perlon staple and 80 percent wool were superior to all-wool suits because of greater elasticity, lower water-absorption, and quicker drying. Neckties, hosiery, ladies' underwear, and suitings have been made from Perlon-wool blends. The principal uses for continuous-filament yarn during the war included parachutes and flare chutes.

No work had been done on the use of Perlon L for airplane tires until the Germans discovered that nylon was used in the tires of American fighter planes and bombers. Extensive analytical reports indicated that the Reich Government had ordered a thorough investigation of the tires from captured fighters and bombers of the American Air Forces.

Attempts to use regular Perlon L having a dry tenacity of 4.5 grams per denier and a dry elongation of 20–21 percent were not successful because of inflation growth. As a result of the urging of the

Luftwaffe, attempts were made to raise the strength and reduce the elongation, and a yarn having a 7-grams-per-denier tenacity and 14–16 percent elongation at break had been produced. However, this yarn was not entirely successful, for when the war ended only 20 tires had been made from it and the work was still in the developmental stage.

2. Perlon Spinning

In general, the spinning equipment was patterned on that which had originated in the U.S.A. The individual steps followed accepted practice—viz.: chip drying, spinning, pretwisting, stretching, twisting, washing, drying, coning and oiling, and sorting and packing, except that the washing and drying are additional operations made necessary because of the relatively high caprolactam monomer content with Perlon L after spinning. A complete appreciation of spinning and processing performance could not be made since access to the major German installations located in the Russian zone at Berlin-Lichtenburg, Premnitz, and Landsberg could not be gained. The indications are that in these operations German practice was behind current American and British practice. The high waste figures and the proportion of second- and lower-grade material produced at Landsberg strongly support this view. Thuringische Zellwolle, Schwarza, was the only target where we were able to inspect spinning procedure, and here the quality of the yarn produced and the spinning performance were very indifferent.

The only novel departure was the band spinning process, which required the preparation of the polymer in the form of a band of specified dimensions. This was then fed for spinning through a close-fitting, heated, rectangular channel. Denier was not so uniform as it is with conventional grid spinning, and it seemed unlikely that the band process would have been developed further because the technical difficulties could not be solved satisfactorily.

3. Plastics and Films

The general trend in new polyamides is more in the direction of plastics, synthetic leathers, and films rather than of fibers. All the dicarboxylic acids with 2 to 16 carbon atoms and the corresponding diamines have been systematically examined, the work covering the ground previously explored by Carothers and his coworkers. This work has so far failed to yield any substantial developments. The most interesting developments are based upon ketopimelic acid, *p*-phenylene dipropionic acid, tetramethylenediamine, *N*-methyl-bis-(*y*-aminopropyl)-amine and *p,p'*-diamino-dicyclohexyl-methane, usually as partial components of polyamides.

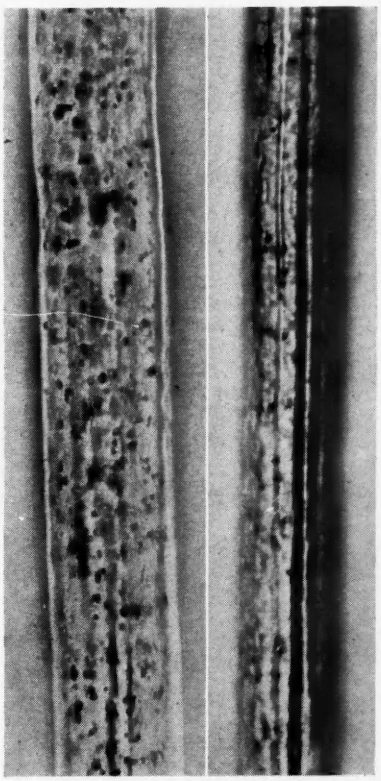
A noteworthy discovery is the polymerization of acetylene to an 8-member-ring hydrocarbon cyclooctatetraene, which may be selectively hydrogenated and then oxidized to suberic acid.

About 1939, I. G. Farben began the production of polyamide films for war uses, principally gas capes and liners for self-sealing gas tanks. There were two general kinds, the first being Lyafol, an interpolymer consisting of 60 percent 66 polymer and 40 percent of caprolactam, known as Igamide 6A.

This was dissolved in an alcohol-water solution and melt-cast to give an unoriented film up to 130 cm. wide and 0.05–0.10 mm. thick. A plasticizer (20–30 percent) was incorporated into the film.

The second type, called Perfol, was 100 percent amino-caprolactam polymer, with or without a plasticizer, which was melt-cast into films up to 150 cm. wide and 0.3–0.4 mm. thick after orientation in both directions. This was superior to Lyafol in sunlight-resistance and resistance to boiling water. It was planned to substitute Perfol completely for Lyafol in gas capes.

Lyafol was made at Wolfen on machines having a



Photographs of cascin fibers which appear in the Report, page 939. Left, Lanital, Snia Viscosa; right, Tiolan, Schwarza.

rated capacity of 500,000 pounds/year but only a fraction of this volume was realized. It was made also at the Kalle & Company plant at Biebrich, which also had a capacity of 500,000 pounds/year.

Contemplated peacetime uses for polyamide films included raincoats, shower curtains, brief cases, school bags, shoe uppers and soles, hospital sheeting, etc. Both unsupported films and fabric coatings were being studied but no final selection had been made.

4. Polyurethanes

During the war years, I. G. Farben at Leverkusen and Höchst have explored the polyurethanes in detail. Technical exploitation was still in an early stage, but a product known as Perlon U (or Igamide U) was under initial development. Two methods were being employed—the reaction of diisocyanates with glycols (Leverkusen), and of glycols with phosgene, followed by interaction with diamines (Höchst). Polymers produced by these two processes show differences, the former process giving polymers more suitable for fibers and bristles, and the latter more suitable for plastics and leather.

Perlon U is produced from 1,4-butane-diol and hexamethylene diisocyanate. The former was, under German conditions, an accessible and low-cost material which was being manufactured on a scale of 125 tons/day at Ludwigshafen from acetylene and formaldehyde as a butadiene intermediate. Hexamethylene diisocyanate was made by phosgenating the carbonate of hexamethylene diamine in dichlorobenzene solution. A 10-tons/month pilot plant had been in operation for about 2 years at Leverkusen but the process was by no means fully established (yield 75–88 percent).

The polymerization process and the plant have some novel features, and the continuous extrusion of the polymer from the polymerization vessel in bristle form as seen at Dormagen, followed by continuous processing all in the one step, is noteworthy.

The diisocyanates are very versatile in their wide range of reaction, and in addition to their use in fibers and bristles they were being evaluated as material for plastics, rubber, lacquer, leather, and adhesives.

In the fiber field, Perlon U is inferior to Perlon T, as it has a much lower softening point, more difficult drawing characteristics, lower tenacity and elongation, and harsher feel. The future of the polyurethanes for textile fibers seems uncertain. They are better adapted for bristles because of the stiffness and low moisture regain. In this connection, the Germans' appreciation was conditioned by their lack of sebacic acid, which gives polyamides better suited for bristles than Perlon T.

The I. G. Farbenindustrie at Ludwigshafen was using the diisocyanate-glycol reaction—hexamethylene diisocyanate and 1,4-butane-diol—which is the basis for Perlon U.

Approximately 25 tons per month of Perlon U fiber were used for parachute fabrics and one ton per month for bristles. The bristles are reported to hold their shape exceedingly well during usage. Polyurethanes melt at 175–180°C, about 50 degrees lower than the corresponding polyamides. This is considered too low to withstand ironing, and they are therefore poor material for textiles. The moisture regain of Perlon U is about one-third the regain of nylon. It had been hoped that Perlon U staple fiber would have more wool-like properties than Perlon L, but the results have been disappointing. Hosiery and underwear knitted from Perlon U had a harsher hand than that made from Perlon L.

5. Vinyl Fibers

The German approach to vinyl fibers has been for the most part through the medium of chlorinated polyvinyl chloride (PeCe fiber). This is a comparatively old development. It was not possible to study in detail all the phases because the time available at the I. G. Farben plants at Wolfen and Bitterfeld and Schkopau was strictly limited because the area was being transferred from American to Russian occupation.

The process for making polyvinyl chloride by Wacker at Burghausen is not impressive because of the comparatively long polymerization cycles involved. Furthermore, some doubt was expressed as to whether this particular type of polymer would be suitable for after-chlorination.

PeCe fiber, because of its low swelling capacity and its resistance to chemical action and bacteria, had important use during the war period in a number of technical fabrics such as capes and hats for the German air force, filter cloths, tarpaulins, tent bottoms, and as a substitute for jute in burlap. A capacity of 327 metric tons per month was available.

Polyvinyl chloride yarns and staple containing 64 percent chloride have been produced from acetone-soluble polyvinyl chloride by the I. G. Farbenindustrie. The fibers were spun at Wolfen from polymer

made at Bitterfeld. For the fiber production, 28 percent solutions in acetone were prepared, filtered hot, and spun into water.

The continuous filament was stretch-spun vertically through water at room temperature on an inverted form of cuprammonium stretch-spinning unit, the yarn being wound up on bobbins. The dilute acetone liquors were used as the spinning bath for PeCe staple and the acetone was recovered from the latter. Three-hundred-denier, 120-filament yarn of 1.8–2.2 grams per denier was produced, with an elongation of 40 percent and a softening point of 100°C.

Staple at the rate of 150 tons per month was produced by forcing the spinning solution through spinnerets, each with 2,500 holes, into 4 percent acetone. Seventy-eight spinnerets were arranged fanwise in 6 baths and the tow was led over 5 sets of rolls, where it was stretched progressively until it was three times its original length.

The tow of 60,000 denier was given a crimp through the I. G. crimper and was cut either as such or as a tow of 200,000 denier by a Gru-gru cutter. The cut staple is dried continuously on a tunnel drier. The staple passes into an opener before baling.

The fabric made from the staple was an inadequate substitute for wool, but was better than viscose or acetate staple in handle.

No evidence was found of vinyl chloride copolymers with vinyl acetate along the lines of vinyon as produced in the United States. There was, however, a substantial production at I. G. Farben at Wolfen of polyvinyl chloride melt-spun monofilm with bristles which had been successfully used in a variety of applications.

Vinylidene chloride was being explored experimentally at I. G. Ludwigshafen as a fiber-forming material, and an 80–100-tons/month pilot plant had been installed there. Vinylidene chloride was chlorinated at I. G. Farben at Schkopau in a 98 percent yield to trichloroethane, and this was converted to vinylidene chloride with aqueous caustic soda at Ludwigshafen. The most suitable polymer for dry spinning (from tetrahydrofuran) contained 7.5 percent ethyl acrylate. Spinning and textile processing at I. G. Farben at Wolfen had been done only on an experimental scale. The general field of application is that in which PeCe fiber is now used, but it was regarded as being a substantial improvement because of its higher softening point and better tenacity and pliability.

Both vinyl and acetate acrylonitrile have been used in the United States in the manufacture of vinyon. No such comparable developments were to be found in Germany. Nevertheless, it should be recorded that processes for making both these monomers for other purposes had been highly developed in Germany. A first-class manufacture of vinyl acetate monomer had been established at I. G. Höchst in a 1,000-tons/month plant which had achieved a very high operating efficiency. Acrylonitrile had originally been produced from ethylene oxide by ethylene cyanohydrin, but this process was far from satisfactory and was being displaced by a more economical synthesis direct from hydrogen cyanide and acetylene, utilizing a liquid-phase, cuprous-ammonia chloride catalyst. Substantial pilot plants had been operated at I. G. Leverkusen and Ludwigshafen and it had been the intention to erect full-scale plants of 250 and 600 tons, respectively. Plant yields of 75–80 percent were currently obtained, and it was considered that with greater experience further improvements would be possible.

The most notable feature in the general field of vinyl polymerization technique was the use of a combination of oxidizing and reducing agents as the catalyst system. This greatly accelerated polymerization as well as increased the molecular weight of the resulting polymers.

The whole approach to the problem of utilizing vinylidene chloride as a basis for synthetic fibers has been to study the copolymerization with small amounts of second components with the object of obtaining the best compromise in properties. The relatively high softening point of polyvinylidene chloride coupled with its lack of thermal stability made this essential. Polyvinylidene chloride is very responsive to the effect of comparatively small amounts of second components (6–15 percent) and exhibits a sharp fall in softening point and an improvement in solubility characteristics within this region.

PeCe 120 (melting point 120°C) is made by the copolymerization of 80–85 percent of vinylidene chloride with 15–20 percent of vinyl chloride. PeCe 120 has been used for the manufacture of bristles by melt-spinning.

In order to produce a polymer suitable for dry spinning from tetrahydrofuran solvent, it is necessary to use 7.5 percent ethyl acrylate instead of 15–20 percent vinyl chloride. This experimental copolymer gives fibers with a softening point of 140–

150°C, which is 60 degrees higher than that of PeCe fiber, together with higher tenacity and greater pliability.

This is an interesting fiber, particularly for specialty industrial applications requiring high resistance to chemical action and fireproof qualities. This fiber should be compared with Saran, which is produced in the United States. The development is still in the experimental stage but is considered to have important potentialities and is expected to yield a product which will supersede PeCe polyvinyl chloride fiber, the latter being quite unsatisfactory because of its low softening point.

6. Polyethylene

Work on polyethylene has been in progress in Germany since 1937–1938. Owing to the severity of bombing at Ludwigshafen, a 20-kilogram-per-hour pilot plant had been dismantled and transferred to Gendorf.

During the war the main interest in polyethylene fibers was as a substitute for jute in the production of burlap bags. It was thought that the resistance of polyethylene to bacteria and rotting would make it an excellent candidate for this use. Its low softening point was not expected to be objectionable for this use.

In spite of the strong claim made by the I. G. Farbenindustrie for the military importance of polyethylene in the electrical field, the military authorities denied their support, either with materials or manpower. It was stated that the military were too late in realizing the full importance of polyethylene to the war effort.

7. Polyvinyl Alcohol

Experimental work on water-soluble polyvinyl alcohol fibers has been done at the Alexander Wacker plant at Burghausen, but these fibers were never produced commercially.

Cellulose Ethers

It was rather disappointing to find that no very fundamental work on cellulose chemistry was being done in Germany. There appeared to be only one aim—to make viscose cheaper and to make it on a larger scale. Very little new work was being undertaken. However, some work on cellulose ethers was in progress at I. G. Farben at Biebrich and Elberfeld. The former is the larger factory, where manufacture takes place on the following scale:

Methyl cellulose (alkali soluble) 10 tons/yr.
Methyl cellulose (water-soluble) 185 tons/yr.
Methyl cellulose (soluble in water and also in organic solvents) 5 tons/yr.
Methyl glycol cellulose (water-soluble) 1,600 tons/yr.
Carboxymethyl cellulose (purified) 600 tons/yr.
Carboxymethyl cellulose—crude (35% carboxymethyl cellulose, 30% salts, 35% water) 9,000 tons/yr.

At Elberfeld the following quantities were made:

Carboxymethyl cellulose (purified) 600 tons/yr.
Benzyl cellulose 180 tons/yr.
Ethyl cellulose 60 tons/yr.

The water-soluble materials are used as emulsifying agents, adhesives, thickeners for foodstuffs, and as detergent aids. The use as adhesives, although developed before the war, might owe part of its success to the shortage of starch. Carboxymethyl cellulose is employed as a detergent aid. It is claimed that when this is added to the washing solution the carboxymethyl cellulose coagulates the dirt, prevents it from settling again on the cloth, gives a brighter fabric, and reduces the soap consumption 50 percent. It is possible that in Germany this reduction in soap consumption was the most important feature of this development, owing to the shortage of fats.

Benzyl cellulose was used as an insulating varnish and also for the preparation of antifouling paints. Ethyl cellulose manufacture had not developed in Germany principally because of the shortage of ethyl chloride.

Conclusion

In the field of applied cellulose chemistry, in which the Germans were outstanding a few decades ago, there has been a noticeable lack of noteworthy accomplishment from the standpoint of getting a more satisfactory fiber. Relatively little effort has been directed toward improving the inherent quality of staple; rather, they have depended upon various after-treatments for improvements in quality.

It appears that the entire objective has been to produce the maximum amount of fiber with the minimum amount of imported raw materials. The Nazi demands upon the German technicians have resulted for the most part in increasing the scale of operation and the development of massive equipment units.

Mechanical Properties of Textiles*

VI. A Study of Creep of Fibers

H. D. Holland,[1] George Halsey,[2] and Henry Eyring[3]

Contribution from The Textile Foundation and the Frick Chemical Laboratory, Princeton University, Princeton, New Jersey

IN the first paper of this series [1], Section IV-C, the theory of the three-element model in relaxation was developed, and it was applied then to the relaxation of rayon. By a similar process, the three-element creep equation can be developed.

The application of this equation to creep is not so simple as the corresponding application to relaxation. Because of the drastic nature of creep experiments, over periods of time the creep curve is observed to divide into several sections of varying importance (duration); one important section is found to be the three-element creep process under discussion.

A second region of importance corresponds to pure viscous flow as represented by a single dashpot, unrestrained by a spring.

The third region is that of autocatalytic break, where, as the molecular chains break, all of the force concentrates on the remaining ones. This type of behavior is discussed by Tobolsky and Eyring [2]. Because of ultimate failure in a restricted neck of small length, this region is often not apparent, although obviously it must exist in every break experiment.

In connection with the flow of materials undergoing breakage it is apparent that these two modes of breaking are to be distinguished. When in every portion of the sample there is equal flow and hence equal attenuation, the relationship between time and elongation will be nearly a linear one, in the range of small elongation, such as the ones commonly encountered in textile mechanics.

When examined from the standpoint of stability, this apparently simple mode becomes complicated because of the special restrictions necessary to make it stable. The requirement is that when a given cross section changes from S to $S - \delta S$ that its elastic constant k increases an amount δk, so that

$$\frac{S}{S - \delta S} < \frac{k + \delta k}{k}.$$

That is, for the elongation of a material to take place without autocatalytic attenuation in one spot, small cross sections must be stiffer than larger, neighboring ones. Aside from placing restrictions on the original shape of the material, which is here assumed to begin with constant cross section, the material must have one of two physical characteristics. Either it must strain-harden, as in the case of the links of a loosely coiled chain pulling tight, or the surface energy must be an important enough factor in the

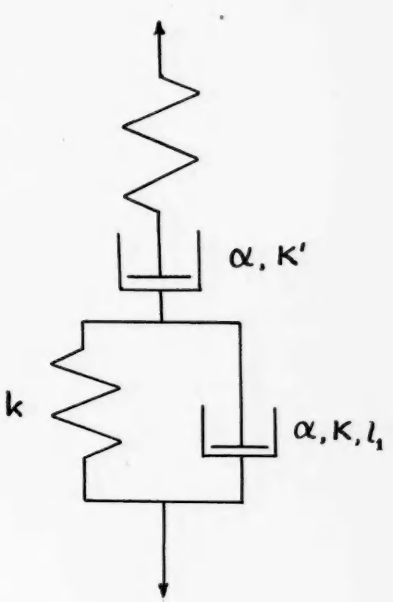


FIG. 1. *The four-element model used in this paper to represent creep.*

* This is the sixth paper in a series, "The Mechanical Properties of Textiles." The previous papers were published in the September and December, 1945, and the January, February, and March, 1946, issues.

[1] This paper constitutes Mr. Holland's senior thesis, partial requirement for the degree of B.A. at Princeton University.

[2] Fellow of the Textile Research Institute, Inc.

[3] Director of Fundamental Research, The Textile Foundation.

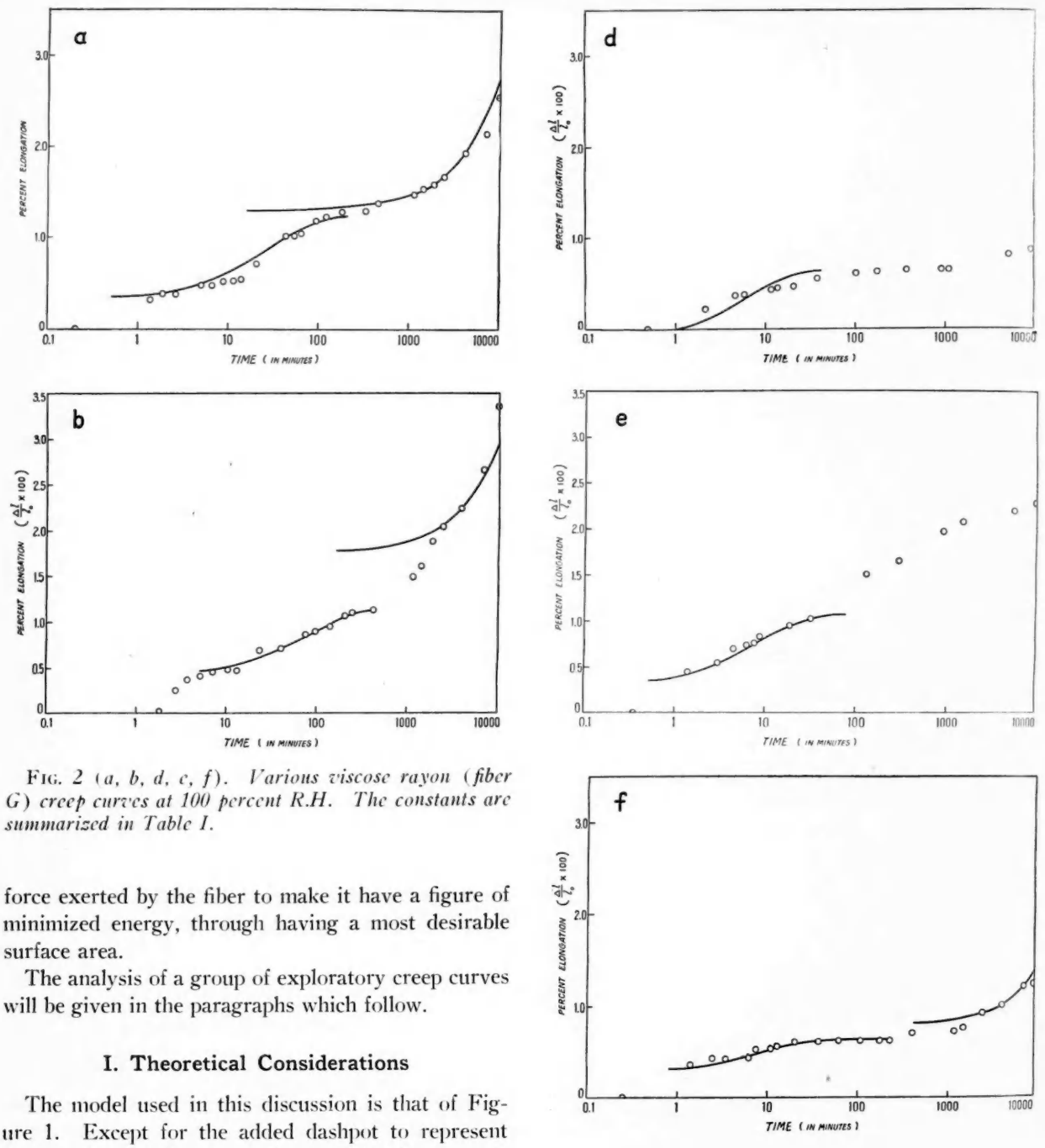

a
PERCENT ELONGATION
TIME (IN MINUTES)
b
PERCENT ELONGATION ($\frac{\Delta l}{l_o}$ x 100)
TIME (IN MINUTES)
d
PERCENT ELONGATION ($\frac{\Delta l}{l_o}$ x 100)
TIME (IN MINUTES)
e
PERCENT ELONGATION ($\frac{\Delta l}{l_o}$ x 100)
TIME (IN MINUTES)
f
PERCENT ELONGATION ($\frac{\Delta l}{l_o}$ x 100)
TIME (IN MINUTES)

FIG. 2 (*a, b, d, e, f*). *Various viscose rayon (fiber G) creep curves at 100 percent R.H. The constants are summarized in Table I.*

force exerted by the fiber to make it have a figure of minimized energy, through having a most desirable surface area.

The analysis of a group of exploratory creep curves will be given in the paragraphs which follow.

I. Theoretical Considerations

The model used in this discussion is that of Figure 1. Except for the added dashpot to represent viscous deformation of an irreversible character, it is equivalent to the model of previous papers of this series. In creep, this dashpot offers no complication, as it merely flows at a constant rate throughout the experiment. Similarly, the open spring assumes its final elongation immediately.

The only elements remaining to be computed are the spring and dashpot included in the Voigt unit of delayed relaxation.

The equations governing these elements are

$$\frac{dl_1}{dt} = K \sinh \alpha f \quad \text{for the dashpot}$$

and

$$F - f = kl_1 \quad \text{for the spring.}$$

They yield

$$-\frac{1}{k}\frac{df}{dt} = K \sinh \alpha f,$$

TABLE I. VISCOSE RAYON FIBER G AT 100% R.H.

Experiment	Time conditioned (days)	Load $\times 10^9$ (dynes per sq. cm.)	l_∞ (%)	t_1 (min.)	$\alpha \times 10^{-9}$ (sq. cm. per dyne)	$k \times 10^{11}$ (dynes per sq. cm.)	$K \times 10^{-4}$ (per sec.)	V_h (cubic Å.)	$\sqrt[3]{V_h}$ (Å.)	$\Delta F^{\ddagger}$ (calories)
a	1	0.671	1.00	28.0	2.5	0.67	13.8	201	5.9	21,240
b	1	0.647	0.725	62.0	2.9	0.89	9.2	228	6.1	21,620
c	3	0.561	0.51	66.0	3.2	1.1	6.4	256	6.4	21,835
d	2	0.548	0.66	4.5	7.3	0.83	1.9	585	8.4	21,690
e	2	0.651	0.70	7.0	2.8	0.93	8.8	220	6.0	21,610
f	1	0.541	0.34	6.6	2.2	1.6	7.9	173	5.6	21,490
g	11	1.106	0.57	4.2	2.5	1.9	3.4	202	5.9	22,100
h	2	0.601	0.753	0.26	2.6	0.79	8.6	207	5.9	21,540
i	11	0.623	0.96	2.8	5.6	0.65	3.4	449	7.7	22,610
j	11	2.31	1.52	46.0	2.5	1.5	1.5	198	5.8	22,660
k	11	1.035	1.03	6.6	4.1	1.0	2.3	326	6.9	22,390

an equation identical with that of the treatment of relaxation in Paper I [1].

Then, this equation was integrated to yield the result

$$\log\left(\frac{\tanh\frac{af}{2}}{\tanh\frac{a}{2}}\right) = t\log\left(\frac{\tanh\frac{a}{4}}{\tanh\frac{a}{2}}\right).$$

Since

$$f = F - kl_1,$$

or in reduced units

$$f_{\text{red}} = 1 - l_1,$$

this equation becomes

$$\log\left\{\frac{\tanh a(1-l_1)}{\tanh\frac{a}{2}}\right\} = t\log\left\{\frac{\tanh\frac{a}{4}}{\tanh\frac{a}{2}}\right\},$$

where the conditions of reduction are

$$\begin{aligned} \text{when } t &= 0, & l_1 &= 0, \\ t &= 1, & l_1 &= \tfrac{1}{2}, \\ t &= \infty, & l_1 &= 1. \end{aligned}$$

With these considerations, the fitting method of Paper I becomes immediately available.

In treating the experimental curves it is merely a convenient approximation to regard the creep curve as consisting of separate sections depending upon various relaxations.

It also is convenient to describe as transition regions those relaxations that are too brief to be important or too involved to be separated. As more data are accumulated, these procedures will be discarded for more exact ones.

II. Experimental Method

The creep experiments were performed in test tubes containing a one-hole rubber stopper with a glass rod to support the fiber. The fiber was attached at its upper end to the rod by means of de Khotinsky cement. For experiments extending over long periods of time, occasionally flow was detected in the cement. This was obviated by selecting especially hard sticks of cement. In extreme cases the use of the plastic Lucite, which is very hard, was found to be necessary. Its higher melting point made damage to the fibers something to be considered, and for a fiber like wool it was clearly not suitable. It seemed quite satisfactory with cotton.

A light metal hook, sufficient to keep the fiber straightened out, was fastened similarly to the lower end of the fiber. The proper concentration of sulfuric acid in the bottom of the test tube regulated the humidity, and the fiber was allowed to condition for a period of time (from a day to a few weeks). The length was then measured. After the fiber had been conditioned the stopper was removed and a glass hook of proper weight was attached to the fiber. The creep curve was then prepared by observation, using the cathetometer.

III. Experiments on Viscose Rayon

The bulk of the experiments were performed on a viscose rayon, du Pont fiber G.*

Figure 2a is one of the curves obtained during the course of the experiments. The dotted points are experimental; the two lines are theoretical. The

* This fiber was furnished us through the kindness of Dr. W. E. Roseveare of the du Pont Company.

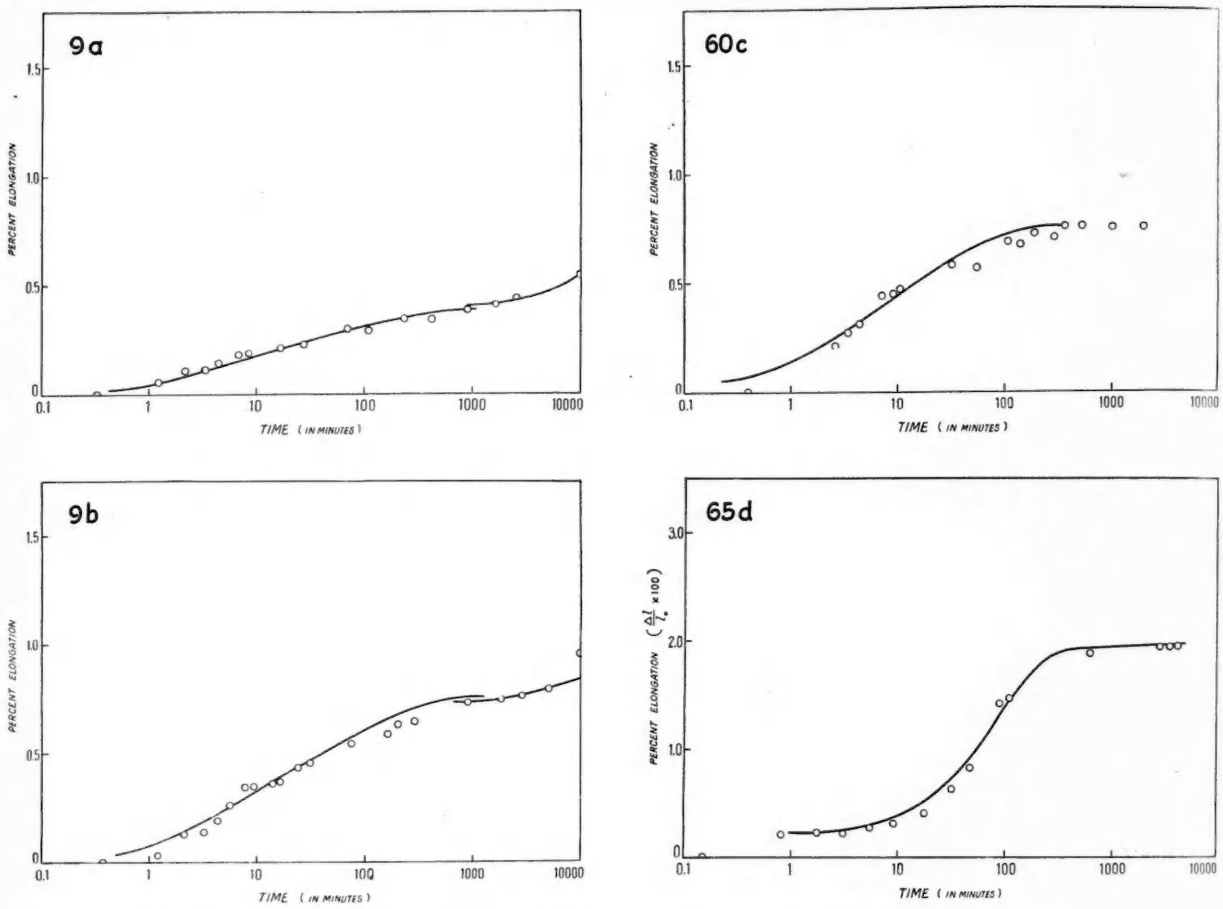
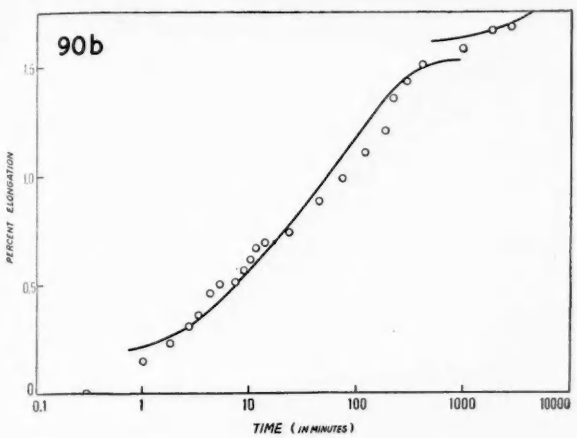
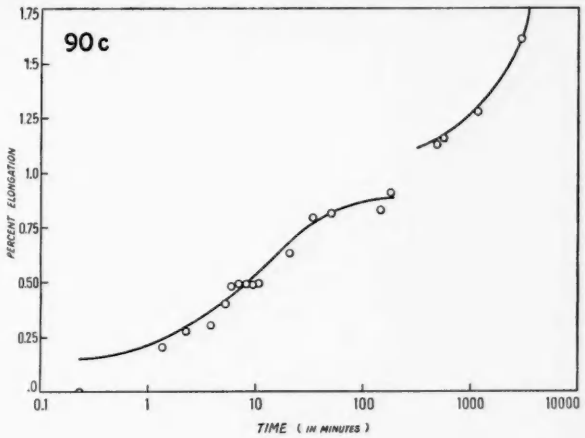


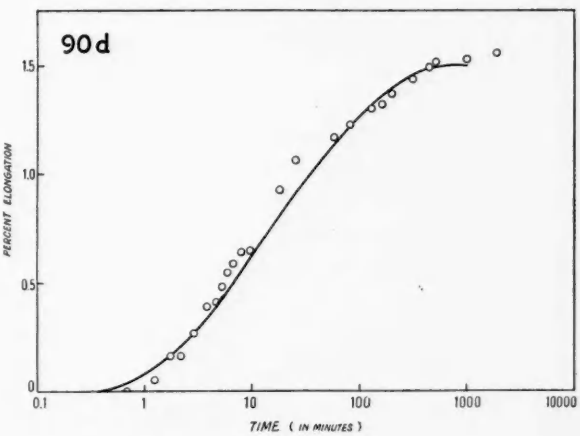
FIG. 3 (*Includes 90b, 90c, and 90d, right*). *Viscose rayon (fiber G) creep curves at varying humidities (Table III).*

TABLE II. VISCOSE RAYON FIBER G

Experiment and relative humidity	Time conditioned (days)	Load $\times 10^9$ (dynes per sq. cm.)	l_∞ (%)	t_1 (min.)	$\alpha \times 10^{-9}$ (sq. cm. per dyne)	$k \times 10^{+11}$ (dynes per sq. cm.)	$K \times 10^{-4}$ (per sec.)	V_h (cubic Å.)	$\sqrt{V_h}$ (Å.)	$\Delta F^{\ddagger}$ (calories)
65a	¾	1.08	0.64	15.0	2.5	1.7	4.7	185	5.7	22,510
65b	16	2.09	2.6	15.0	4.0	0.8	0.44	317	6.8	23,590
65c	16	2.32	1.32	60.0	3.3	1.8	0.31	262	6.4	23,680
65d	½	0.525	1.70	59.0	1.9	0.3	8.6	146	5.3	20,540
65e	2	2.31	0.78	1.4	1.6	2.96	2.4	127	5.0	22,390
90a	¾	1.11	1.25	200.0	4.9	0.893	1.1	393	7.3	23,170
90b	¾	1.38	1.40	295.0	3.4	0.995	2.2	274	6.5	22,534
90c	8	1.14	0.80	8.0	3.4	1.424	2.2	272	6.5	22,530
90d	8	2.11	1.57	13.8	2.4	1.34	2.1	191	5.8	22,350
60a	¾	1.17	0.90	325.0	3.2	1.3	3.0	256	5.8	22,310
60b	¾	1.05	0.75	54.0	4.3	1.4	1.36	344	7.0	22,960
60c	8	1.04	0.76	6.8	4.0	1.37	1.1	368	7.2	23,130
60d	8	1.12	0.59	10.0	3.9	1.9	1.2	309	6.8	23,030
9a	¾	1.45	0.37	13.0	4.5	3.9	0.25	354	7.1	23,830
9b	¾	2.31	0.77	17.0	2.7	3.5	0.22	218	6.0	23,770







constant, α, was deduced from the experimental points. The upper curve, when not plotted on semi-logarithmic paper, represents a straight line, and is determined by the experiment, as will be explained later in this paper.

The first reading was obtained at $t = 20$ seconds. The elongation for this time was set at zero since an accurate measure of elastic elongation was not available. The interval between 20 seconds and 2 minutes is here characterized by a gradual decrease of slope. This is the transition region from the elastic portion, where elongation is very rapid, to the creep mechanism which begins to act only after a certain period of time has elapsed. The fact that such a transition region does exist is evidence that beside the main dashpot in the model others of very much smaller relaxation times exist in the fiber.

The period from 2 to 200 minutes is dominated by reversible creep, as is seen by the fit of the theoretical curve to the experimental points. As in Figure 2—and this is generally true for all these fibers—the experimental points coincide approximately with the theoretically calculated curve.

From 200 to 300 minutes the transition from primary (reversible) to secondary (irreversible) creep is occurring. The experimental points here begin to deviate sharply from the theoretical curve and tend

TABLE III. SECONDARY CREEP

Experiment	Load $\times 10^9$ (dynes per sq. cm.)	Humidity (% R.H.)	Slope per 10^8 min.	$K' \times 10^{-7}$	$\Delta F^{\ddagger}$ (calories)
e	0.652	100	4.8	8.0	25,600
a	0.671	100	15.0	25.0	24,900
f	0.531	100	3.8	6.3	25,600
b	0.561	100	12.5	21.0	25,000
g	1.105	100	8.1	13.5	25,200
90a	1.111	90	6.0	10.0	25,800
90b	1.38	90	3.9	6.5	25,800
k	1.03	100	18.0	30.0	25,000
65e	2.30	65	3.8	6.3	25,400
9a	1.45	9	1.61	2.7	26,500
9b	2.31	9	1.58	2.63	26,200

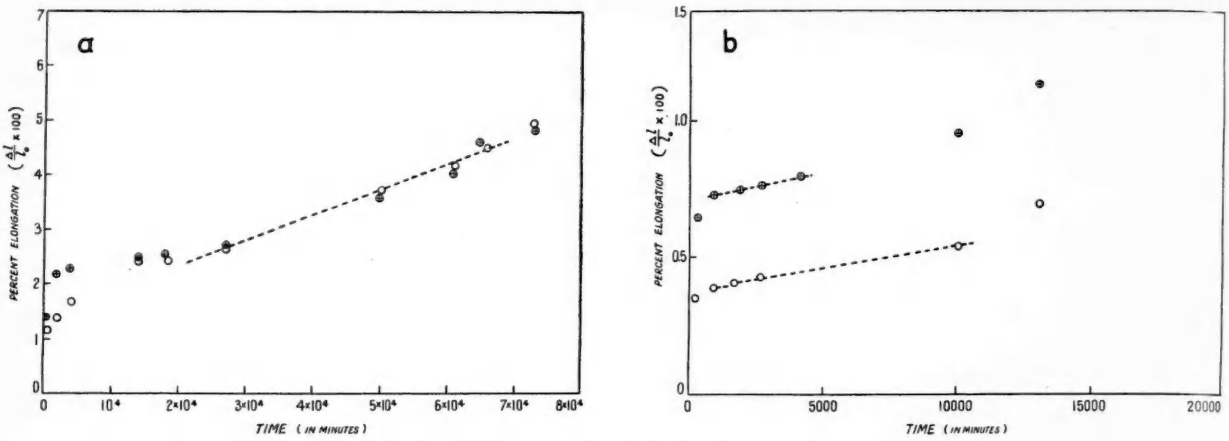


FIG. 4. *Linear creep curves for rayon (fiber G) showing the secondary dashpot.*

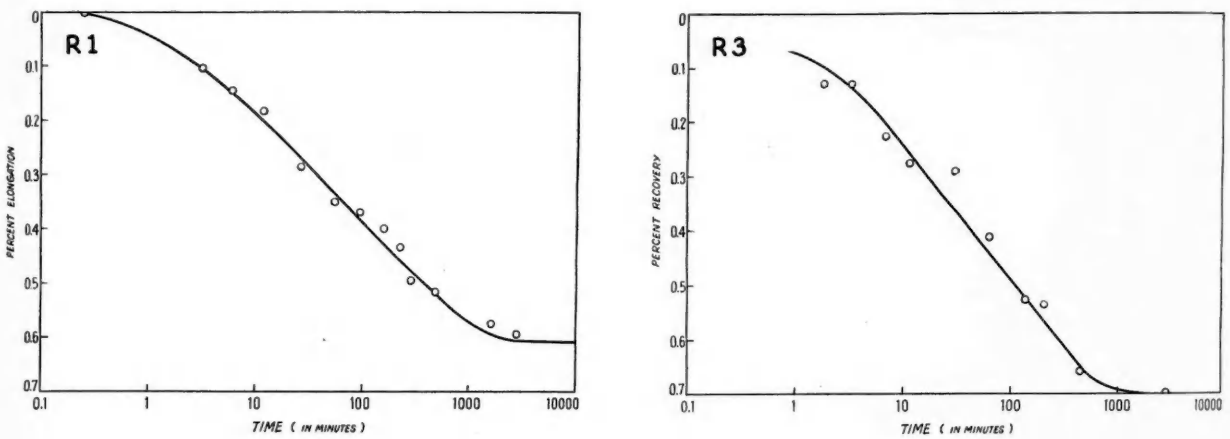


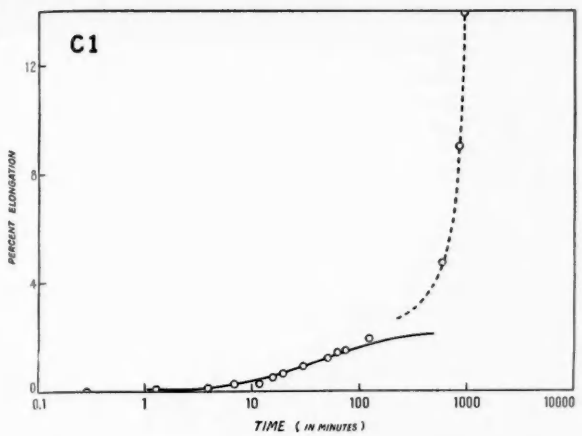
FIG. 5. *Recovery of rayon (fiber G) (Table IVa).*

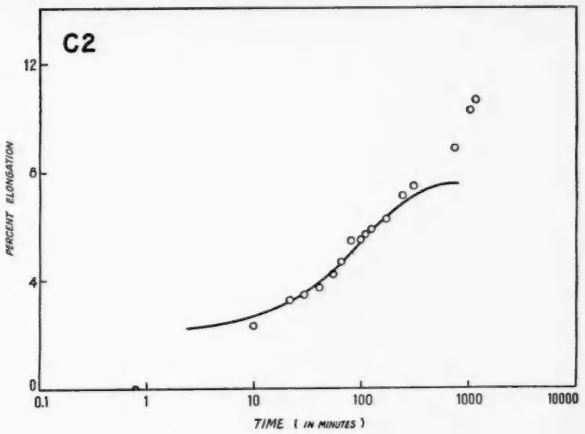
to follow more and more the straight-line pattern indicated above. In this particular experimental run this transition region is negligible. In other cases, some of which will be noted later in this discussion, the region of transition was much extended. In the model this means that the relaxation times of the two dashpots involved are more nearly equal. It seems that sometimes the fiber never reaches the theoretical breaking stage, but always remains in the transition portion.

Finally, the time interval from 300 to 10,000 minutes was almost entirely dominated by secondary creep, as witnessed by the excellent fit with a straight line.

TABLE IVa. CREEP RECOVERY

Experiment	$F\times10^{9}$ (dynes per sq. cm.)	l_f (%)	t_1 (min.)	$\alpha\times10^{-9}$ (sq. cm. per dyne)	$k\times10^{11}$ (dynes per sq. cm.)	$K\times10^{-4}$ (per sec.)	$\sqrt[3]{V_h}$ (Å.)	$\Delta F^{\ddagger}$ (calories)
R–1	0.535	0.651	40	15.9	0.660	0.256	10.6	24,740
R–2	0.695	0.425	56	13.6	1.632	0.101	10.1	25,200
R–3	0.791	0.712	30	10.2	1.11	0.467	9.3	23,210





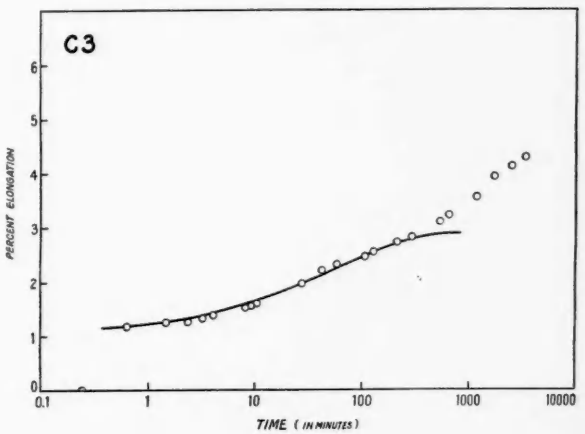


FIG. 6 (*C1, C2, C3*). *Creep curves for cotton (Table V).*

TABLE IVb.

Primary elongation (%)	Secondary elongation (%)	Creep recovery (%)
0.315	0.588	0.441
.563	0.319	.425
.304	3.14	.27
.88	2.54	.74
.268	2.26	.585
.36	0.46	.35
.218	2.27	.67
1.75	0.0	0.897

The detailed analysis of creep phenomena is now considered. After it was ascertained that there usually existed a region where the theory developed at the beginning of the paper could be applied, experiments were carried out further to elucidate the properties of fibers on the basis of this theory. The variables considered were the weight attached to the fiber and the humidity at which the stretching of the fiber was carried out. The results of these experiments, which will be cited briefly here, are summarized in Table I and illustrated in Figure 2. These experiments all were performed at 100 percent humidity and at about 25°C.

When the variation of k, the spring constant, with load was investigated, no definite trend was noticed. This shows that as far as loading is concerned the springs are Hookean in the range of the forces used. This verifies an assumption made in the model.

From an analysis of the data, the volume of the flow-unit could be calculated. It was found that, although it was not always the case, the size of the flow-unit usually decreased more or less regularly

TABLE V. DATA FOR COTTON

Experiment	Sample	Load $\times 10^9$ (dynes per sq. cm.)	l_∞ (%)	t_1 (min.)	$\alpha \times 10^{-9}$ (sq. cm. per dyne)	$k \times 10^{11}$ (dynes per sq. cm.)	V_h (cubic Å.)	$\sqrt[3]{V_h}$ (Å.)	$\Delta F^{\ddagger}$ (calories)
C–1	V–10	0.96	2.1	27	3.2	0.45	256	6.3	22,935
C–2	V–10	0.368	5.4	75	4.37	0.068	348	7.0	23,285
C–3	V–11	0.631	1.75	34	7.65	0.30	610	8.5	22,590
C–4	V–10	0.778	2.16	100	5.78	0.36	460	7.7	22,470

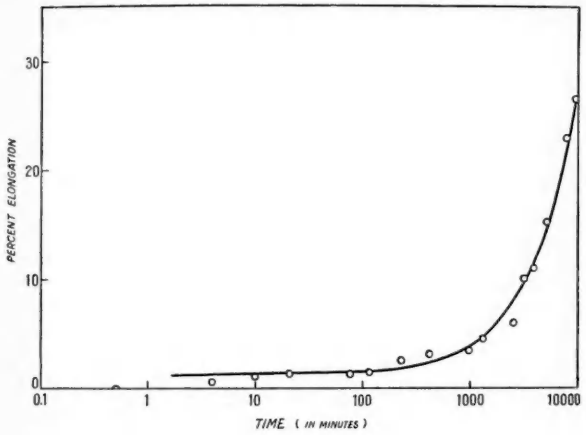


FIG. 7. *Secondary creep in cotton.*

with increased load for the same humidity and same length of time of conditioning.

The specific rate constant K and, thus, the activation free energy $\Delta F^{\ddagger}$ of the creep process showed no decided trend with increased load.

Experiments were carried out at varying humidities. The results are summarized in Table II.

As shown in Figure 3, the shape of the resulting creep curves is much the same at all humidities. Nevertheless, several features are quite different. The portion of time allotted for the transition from elastic to creep deformation becomes successively smaller as the humidity is decreased. This indicates that the dashpots of small relaxation times become frozen at low humidities. In other words, at low humidities a large amount of activation energy is required to make the units associated with these dashpots slip. It is a logical conclusion that at high humidities, where the water content of the fiber is much higher than at low humidities, the water molecules lubricate the slip of sections of molecules past each other by a separation of the chains and a consequent decrease in the secondary valence forces which give rise to these energy barriers and associated activation energies.

A further investigation shows that at considerably lower humidities (below 10 percent) the spring constant, k_1, increases considerably. The fact that the activation energies in each case are higher after a long period of conditioning is clear in all cases except that of 100 percent humidity.

For instance, the fact that for the case of 60 percent humidity $\Delta F^{\ddagger}$ after one day of conditioning was 22,640 calories, whereas after eight days it was 23,080 calories, definitely shows that equilibrium between the fiber and its surroundings is not attained until an appreciable time has elapsed. In the case of 100 percent humidity the reverse was to be expected since before conditioning the fiber had been kept in a room where the humidity was close to 65 percent.

In several of the above-mentioned instances evidence for a second dashpot in the creep mechanism is quite strong. This is shown by a nearly flat region between $t = 5$ and $t = 12$ minutes.

When the data of various creep experiments were plotted, a straight-line relationship was found to hold over long ranges of time in the experiment, indicating a secondary, open dashpot. Figure 4A shows the results of the longest creep experiments performed. After the rapid initial elongation the slope of the line remained essentially constant over a period of five weeks, at the end of which time the experiment was discontinued. In many other cases similar phenomena were observed. The existence of a secondary dashpot, therefore, is indicated. Furthermore, in Figure 4B the set of data indicated by o shows that the slope in many cases tends to increase with time. Since these experiments were carried out at elongations of the fibers of usually less than 10 percent, the accelerating factor of the uniform decrease of cross section due to lengthening is still negligible. The acceleration, therefore, must have been due to rapid slippage at one or more weak points in the fiber.

Table III summarizes the data on the secondary dashpot. To determine fully the mechanism and the flow process, two constants, K' and α', have to be found. The method of analysis allowed the absolute determination of one of these. Therefore, it was assumed that the units which were flowing were of the same dimensions as those involved in primary creep, so that, since $\alpha = V'h/2kT$, α would be the same for the two processes:

$$\alpha' = \alpha.$$

When these values of α' were substituted into the rate equation for the dashpot, K' was determined.

When the load is removed, there is a sudden elastic contraction followed by delayed creep recovery. Several creep-recovery curves were prepared (Figure 5) as can be seen from the values in Table IVa. The recovery of rayon fiber G is restricted mainly to elongation of primary creep. Nevertheless, it seems that some of the apparent secondary creep also

TABLE VI. DATA FOR WOOL AT 100% R.H.

Experiment	Force $\times 10^9$ (dynes per sq. cm.)	l_∞ (%)	t_1 (min.)	$\alpha \times 10^{-9}$ (sq. cm. per dyne)	$k \times 10^{11}$ (dynes per sq. cm.)	$K \times 10^{-4}$ (per sec.)	V_h (cubic Å.)	$\sqrt[3]{V_h}$ (Å.)	$\Delta F^{\ddagger}$ (calories)
W-1	0.108	0.551	20	25	.21	2.74	1,980	12.6	22,470
W-2	0.407	0.68	21	6.6	.60	4.34	527	8.08	22,040
W-3	0.362	0.65	49	2.3	.55	20.4	201	5.86	21,910
W-4*	1.762	1.15	12	6.9	.15	27	551	8.20	22,340

* Recovery curve.

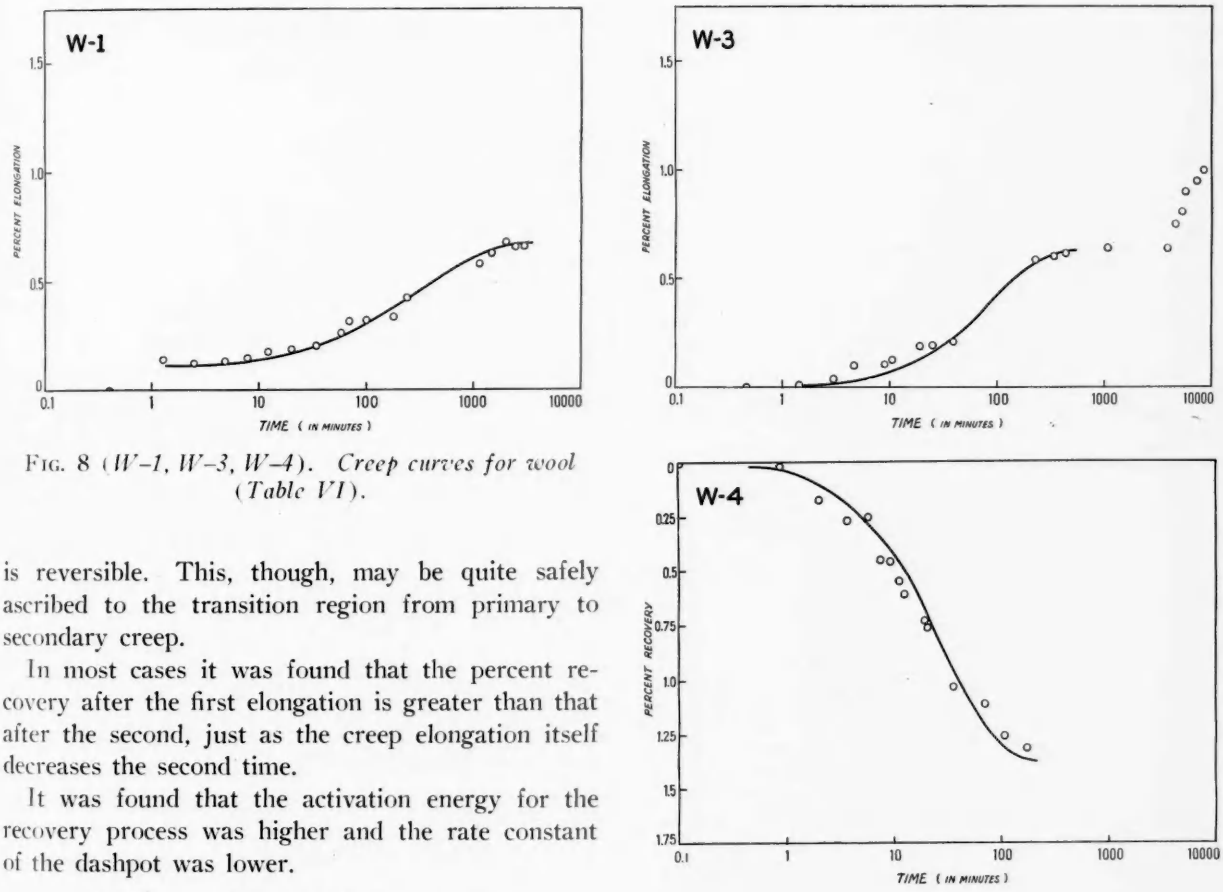


FIG. 8 (*W-1, W-3, W-4*). *Creep curves for wool (Table VI).*

is reversible. This, though, may be quite safely ascribed to the transition region from primary to secondary creep.

In most cases it was found that the percent recovery after the first elongation is greater than that after the second, just as the creep elongation itself decreases the second time.

It was found that the activation energy for the recovery process was higher and the rate constant of the dashpot was lower.

$$\text{Average} \quad K = 0.276 \times 10^{-4} \text{ sec}^{-1}$$
$$\text{Average } \Delta F^{\ddagger} = 24{,}380 \text{ calories}$$

Furthermore, the average slip volume was found to have an average length on a side of 10 Å., a value which is appreciably larger than the constant for the corresponding creep experiments.

IV. Experiments on Cotton

Since cotton, like rayon, possesses the fundamental structure of cellulose, a similar behavior of the two types of fibers was to be expected. On the other hand, the greater irregularities of the cotton might lead one to suspect a greater number of irregularities in the resultant creep curves. The first supposition was found to be correct. This is clearly shown in Figure 6.

A closer analysis of cotton creep curves, which were fitted in the same way as those for rayon, shows that the region marking the end of primary and the beginning of secondary creep is rather indistinct. In fact, in some cases the only evidence of such a transition was a change of slope in the graph. This is likely to introduce errors into the analysis of creep curves for cotton.

In some cases (Figure 7), the region corresponding to creep is absent or nearly absent, as is seen from the fact that the creep curve can be fitted quite successfully by a simple dashpot. This indicates that secondary creep is more prominent for cotton than for rayon at the same load.

The fact that the percentages of elongation in creep experiments conducted with cotton were so much larger than those found for rayon is quite striking. Table V is a summary of the constants for creep of cotton.*

From Table V it is clear that the values of all the constants are approximately the same as those for rayon. The volume of the slip-units are of the same magnitude and the rate constant for the dashpot has analogous values. The spring constant, k_1, is seen to be somewhat small as compared to that for rayon elongated under the same conditions—one day of conditioning at 100 percent humidity.

V. Experiments on Wool

The last type of fiber investigated was wool. Since this is an animal fiber as contrasted to the vegetable fibers considered before, a different set of mechanisms might have been expected.

* The cotton used was supplied us by the Pacific Mills.

Table VI gives the constants for an analysis of the curves shown in Figure 8.

The volume of the unit cell is taken as an approximation for the moving unit (V_h). According to Astbury [3] this unit for keratin has the dimensions $3.5 \times 4.5 \times 9.5$ Å. in the unstretched form, giving 150 Å.^{3} for V_h. Whether or not the unit cell for the type of wool used has actually this volume is not quite certain.

The constants themselves show that, whereas the spring contant, k, is approximately equal to that of rayon, the viscous element characterized by $\Delta F^{\ddagger}$ and K has different values.

The creep recovery tests indicated, as was to be expected, that for the loads used creep was 100 percent recovered, in contrast with rayon, for which this was not found to be the usual case.

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Devices for Producing Artificial Rainfall for Tests of Water-Repellent and Waterproof Materials*

M. I. Landsberg† and F. J. Moody‡

This article is one of a series of technical papers dealing with the principal phases of the research, development, and production problems of the Army's water-repellent clothing. These publications will appear in early issues of various journals in the fields of textiles and garments, and will emanate from the laboratories and organizations participating in the Quartermaster Corps research and development program.

Abstract

The development of devices for the production of simulated rainfall was initiated by the Quartermaster Corps for use in the evaluation of water-repellent characteristics of textile fabrics and garments. In order to simulate natural rainfall, intensities, drop sizes, and drop velocities similar to those found in natural rainfall must be produced. Three nozzles furnishing approximately 0.1, 1, and 3 inches of rainfall per hour, respectively, have been developed to accomplish this purpose.

Measurements have indicated that the sizes of the drops produced by the three nozzles agree quite well with the sizes of the drops found in natural rains of similar intensities. Correct positioning of the nozzles with respect to height and angular orientation has made it possible for the drops to attain terminal velocity before striking the test subject. Drop sizes were measured by catching the drops in flour, drying and sorting the pellets formed by the drops through a series of standard sieves, and determining the pellet size distribution by means of a calibration curve relating pellet size to drop size. Drop-velocity measurements were made by photographing a waterdrop at varying heights of fall, using stroboscopic light as the source of illumination. Intensity measurements were made by catching the falling drops in cylindrical containers spaced at 2-foot intervals throughout the test area, measuring the volume of water collected, and calculating it in terms of inches of rainfall per hour.

A number of variables were studied in the development work, such as water pressure, nozzle height, design, and orientation with respect to the horizontal.

Early Work on Artificial Rainfall

There has long existed a need for a convenient and reliable method of determining the efficacy and durability of apparel fabrics in protecting the American soldier from the discomforts of rain. Laboratory tests have been deemed inadequate in that they are capable of determining only whether one fabric is superior to another in a given characteristic. The relationship of measurements on laboratory apparatus to the length of time which a garment made from the test fabric will keep the wearer dry in a rainstorm had hitherto not been established.

The most direct means of evaluating water-repellency would be the testing of garments in actual rainfall; however, it does not always rain during the periods when testing is to be accomplished. When it does rain, the rate of rainfall is rarely uniform for long enough periods to permit direct comparisons between fabrics. Variables such as wind velocity and air and water temperature make controlled testing difficult under actual rainfall. To provide the necessary control, investigation of devices to produce artificial rainfall was proposed by the Military Planning Division of the Office of The Quartermaster General.

Considerable work has been reported on attempts to create artificial rainfall. Thorough studies have been made of the characteristics of natural rainfall throughout the world. The Soil Conservation Service [1] worked on methods of creating artificial rainfall in connection with erosion studies. Defant [2],

* Based upon a report of the Research and Development Division, Philadelphia Quartermaster Depot, Lt. Col. F. M. Steadman, Director, to the Military Planning Division of the Office of The Quartermaster General.

† Technologist, Research Laboratory Section, Research and Development Division, Philadelphia Quartermaster Depot.

‡ Consultant to the Office of The Quartermaster General.

Bentley [3], Laws and Parsons [4], Laws [5], and Lenard [6] have contributed valuable information on the physical characteristics of both natural and artificially created rainfall. Applicable experiments of these early investigators have been repeated in this study and are cited in this paper. However, it was felt that each investigator did his experimental work to meet a particular problem; the requirements of the Quartermaster Corps made further work necessary.

Experimental Work for Quartermaster Project

Research during the war has been influenced to a great extent by the urgent need for immediate answers, as well as by the lack of personnel and equipment. In keeping with these conditions the first logical step in the project on development of devices for creating artificial rainfall was to survey available sources of fire-fighting and garden-type nozzles. Commercial catalogs revealed numerous promising devices, samples of which were evaluated at the Philadelphia Quartermaster Depot.

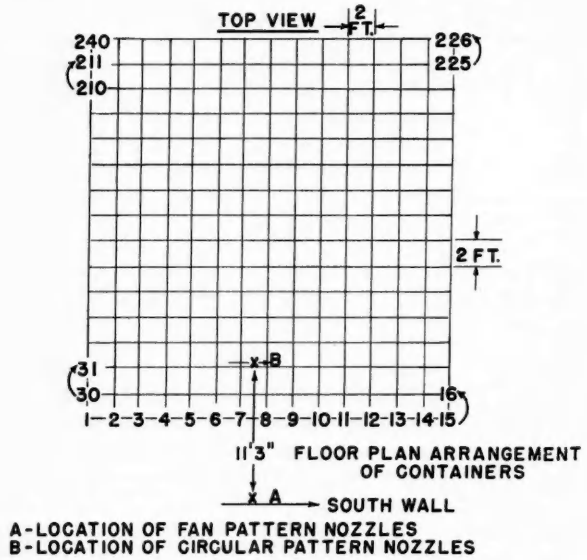
Intensity Measurements

The study of rainfall intensities furnished by the test nozzles was undertaken first, to eliminate in the early stages of the project those devices which furnished too much or too little water, and those which provided uneven distributions of rainfall or excessive amounts of mist. The nozzles were connected at varying heights and at pressures deemed suitable for the individual types tested. The floor of the testing site was laid off in 2-foot squares, as shown in Figure 1.

Cylindrical containers, identified by location numbers, were used as rain gages. The rain pattern of each nozzle was the deciding factor in determining its location in the test room. Those nozzles which threw the water forward in a fan-shaped pattern were placed against the wall of the test room; those creating a circular pattern were connected to a horizontal pipe extending approximately 11 feet into the center of the room. A solid wooden platform (Figure 1) was constructed to service and regulate the nozzles during the testing program.

Upon completion of the preliminary testing, three nozzles were retained for further experimentation—two baffle types and one rotary type.

The first flat baffle nozzle which furnished a fan-shaped pattern of approximately 1 inch of rainfall per hour was experimented with, and the factors



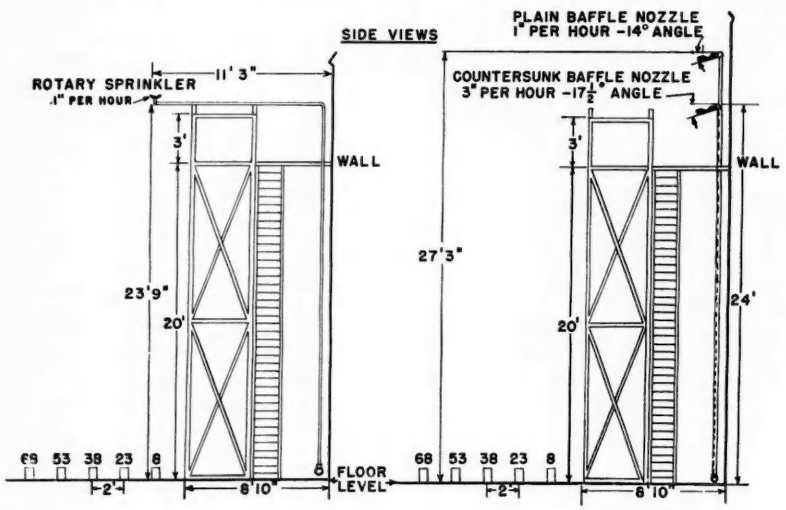


FIG. 1. *Plan of test site in Philadelphia Quartermaster Depot Rain Room.*

leading to improved distributions at the desired intensity level were investigated. Systematic variation of water pressure, baffle angle, height of nozzle, diameter of orifice, and angle of nozzle with respect to the horizontal led to recommendation of the nozzle (Figure 2) and the constants cited in Table I.

TABLE I. DIMENSIONAL AND OPERATING CONSTANTS FOR THE FLAT BAFFLE NOZZLE FURNISHING FAN-SHAPED RAIN PATTERN (1 INCH PER HOUR)

Water pressure (lbs./sq. in.)	30
Water flow (gals./hr.)	425
Baffle angle (degrees)	49
Angle of nozzle to horizontal (degrees)	−14
Height of nozzle (ft.)	27¼
Diameter of orifice (in.)	0.25

Of the 35-by-35 foot area wet by the spray, a test area was selected 16-by-16 feet (Figure 3). In this area 79 percent of the intensity values lie between the limits 0.82 inch and 1.02 inches per hour; that is, the average intensity was 0.92 ± 0.1 inch per hour.

Retest of the 1-inch-per-hour nozzle gave evidence of the reproducibility of results when all conditions cited (Table I) were maintained. Machining of the nozzles was found to be critical, for a number of specimens had to be discarded because of significant differences in spray distribution in the area designated for testing purposes.

The second baffle nozzle was tested with successive variations in the constants of design and operation. An additional feature, the countersunk baffle, was introduced (Figure 4). This provided improved uniformity of distribution of intensities at a 3-inch-per-hour level.

Final recommendations for nozzle constants are listed in Table II.

The area designated as the test area for the 3-inch-per-hour rainfall was 18-by-18 feet, with 84 percent of the intensity values falling between 2.9 and 3.3 inches per hour (Figure 5); that is, the average intensity was 3.1 ± 0.2 inches per hour.

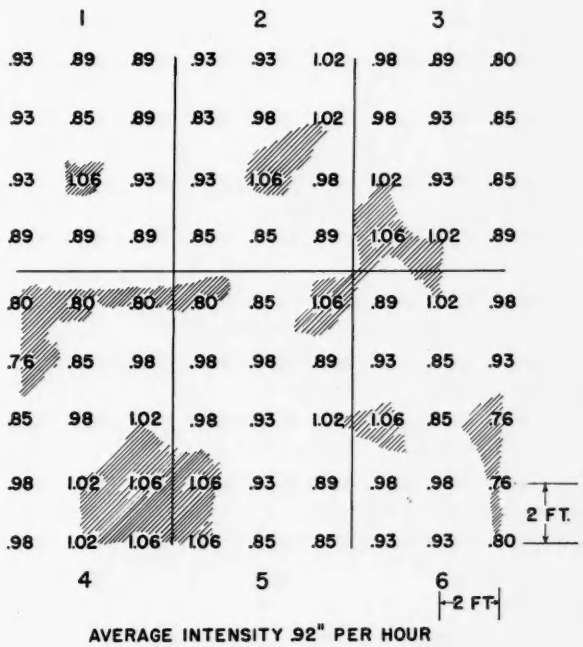


FIG. 3. *Intensity distribution for flat baffle nozzle furnishing 1-inch-per-hour rainfall. Shaded areas indicate portions of test site not included in the 0.82-to-1.02-inch-per-hour rainfall range. Numbered areas shown by black line indicate location of drop-size measurements.*

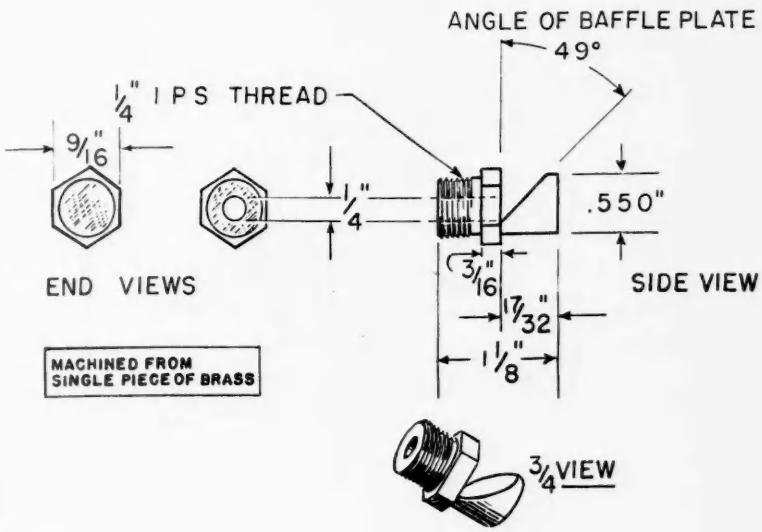


FIG. 2. *Flat baffle nozzle. Furnishes 1-inch-per-hour, fan-shaped rain pattern.*

TABLE II. DIMENSIONAL AND OPERATING CONSTANTS FOR THE COUNTERSUNK BAFFLE NOZZLE FURNISHING FAN-SHAPED RAIN PATTERN (3 INCHES PER HOUR)

Water pressure (lbs./sq. in.)	21
Water flow (gals./hr.)	2,600
Baffle angle (degrees)	45
Angle of nozzle to horizontal (degrees)	−17.5
Height of nozzle (ft.)	24
Diameter of orifice (in.)	$\frac{11}{16}$

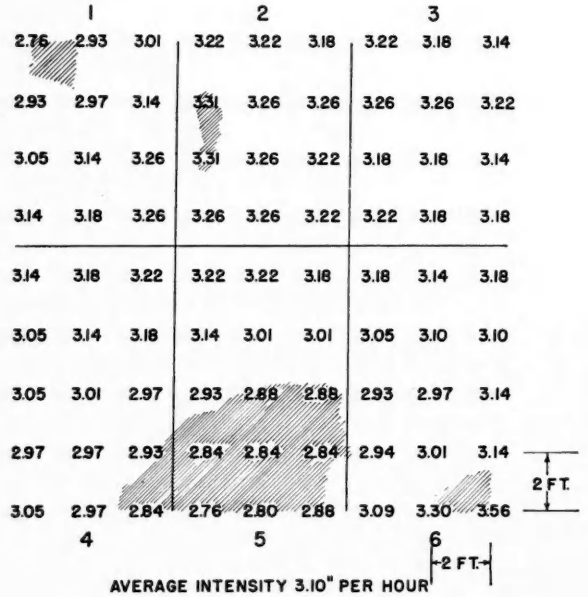


FIG. 5. *Intensity distribution for countersunk baffle nozzle furnishing 3-inch-per-hour rainfall. Shaded areas indicate portions of test site not included in the 2.90-to-3.30-inch-per-hour rainfall range. Numbered areas shown by black line indicate location of drop-size measurements.*

The rotary-type nozzle which produced the 0.1-inch-per-hour rainfall was a commercially available lawn sprinkler. It was found that the minimum intensity obtainable by varying the operating conditions of the sprinkler was 0.2 inch per hour. The orifice of one arm of the sprinkler was soldered shut, thereby reducing the intensity to the desired level of 0.1 inch per hour (Figure 6).

The dimensional and operating constants of this nozzle are shown in Table III.

TABLE III. DIMENSIONAL AND OPERATING CONSTANTS FOR THE ROTARY SPRINKLER FURNISHING CIRCULAR RAIN PATTERN (0.1 INCH PER HOUR)

Water pressure (lbs./sq. in.)	18
Water flow (gals./hr.)	180
Angle of nozzle to horizontal (degrees)	90
Height of nozzle (ft.)	$23\frac{3}{4}$
Size of orifice (in.)	0.5

Of the circular area 30 feet in radius which was wet by the spray, a test area 22-by-14 feet was selected. In this area 79 percent of the intensity values were between the limits of 0.111 and 0.131 inch per hour; that is, the average intensity was 0.121 ± 0.01 inch per hour (Figure 7).

Drop-Size Measurements

The method for determining drop-size measurements was the flour technique described by Bentley [3] and Parsons and Laws [4]. It consisted of catching in flour the drops to be measured, drying and sorting the pellets thus formed, and determining from these the drop-size distribution of the rainfall. The first step of the procedure was the calibration of the flour, since different flours vary in composition.

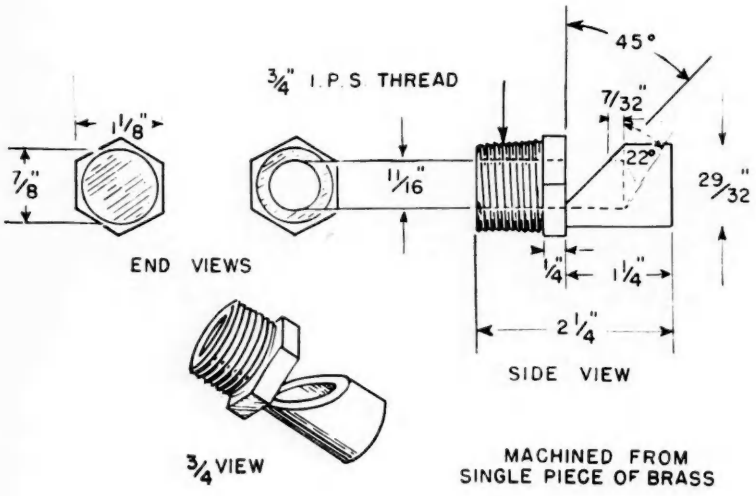


FIG. 4. *Countersunk baffle nozzle. Furnishes 3-inch-per-hour, fan-shaped rain pattern.*

Calibration was accomplished with 11 glass capillary tubes which furnished drops ranging in weight from 0.671 to 15.71 mg. Water was introduced into each tube to a given level and then permitted to flow out. When the rate of flow was constant, a counted number of drops were caught and weighed to establish average drop weight for the given capillary. This procedure was repeated three times for each tube to establish the accuracy of the average drop weight. A counted number of drops were then allowed to fall into a petri dish approximately ¾ inch high and 3½ inches in diameter, filled with uncompacted flour which had been sifted through a 60-mesh sieve and smoothed lightly. The pellets formed by the water striking the flour were allowed to air-dry overnight, then were removed by sifting, and dried for 2 hours at 110°C. The pellets were cooled in a desiccator, weighed, and counted to establish a value of average pellet weight for each capillary tube.

Repeating the procedure for all the capillary tubes provided eleven corresponding values of average waterdrop weight and average flour-pellet weight. These values when plotted indicated a linear relationship between pellet weight and drop weight, or drop size, and established the necessary flour calibration for subsequent measurement of rainfall drop-size distributions (Figure 8).

Drop-size determinations were made in duplicate on the 0.1-, 1-, and 3-inch-per-hour nozzles in the areas indicated by the solid lines in Figures 3, 5, and 7. The procedure was the same as that followed for calibrating the flour except that after drying the pellets were sifted through a series of standard sieves of 6, 8, 10, 14, 20, 28, and 35 mesh to divide the sample into class intervals of pellet size. The pellets of each class interval were weighed and counted to obtain the average pellet weight for each sieve size and to determine, by reference to the flour calibration curve, the average drop size. In this manner a frequency distribution of drop sizes was obtained.

Significant differences in drop-size distribution were noted between the combined areas, 4, 5, and 6, and areas 1, 2, and 3 covered by the 1-inch- and 3-inch-per-hour nozzles (Figures 9 and 10). Further computations of drop-size distributions of these nozzles were therefore based on the combined areas 4, 5, and 6, and the areas 1, 2, and 3. Similarly, areas 1, 3, 4, and 6, and areas 2 and 5 for the 0.1-inch-per-hour nozzle (Figure 11) were combined for purposes of computation.

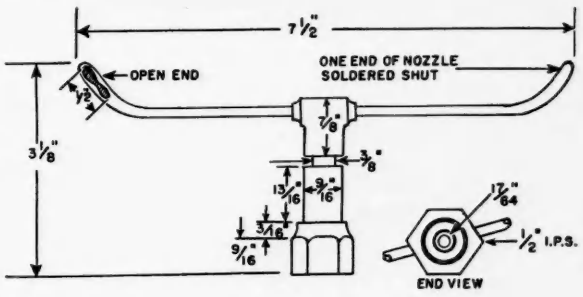


FIG. 6. *Rotary sprinkler. Furnishes circular rain pattern, 0.1-inch-per-hour.*

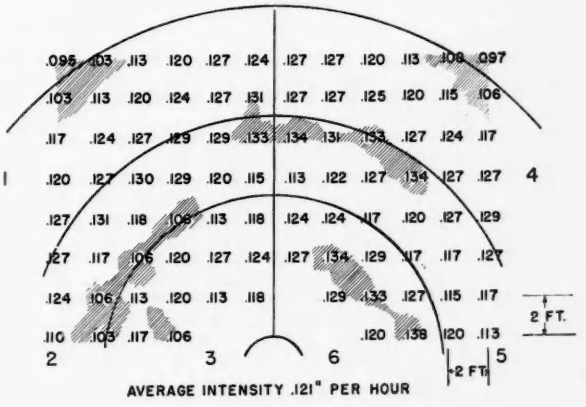


FIG. 7. *Intensity distribution for rotary sprinkler furnishing 0.1-inch-per-hour rainfall. Shaded areas indicate portions of test site not included in the 0.111-to-0.131-inch-per-hour rainfall range. Numbered areas shown by black lines indicate location of drop-size measurements.*

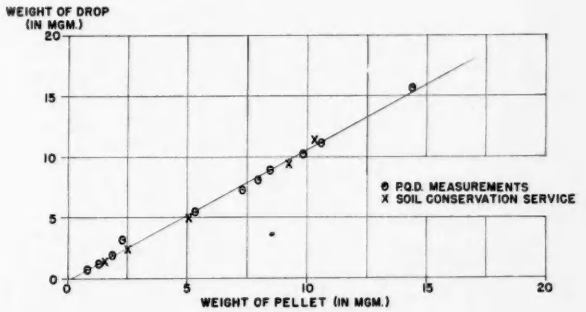


FIG. 8. *Calibration of flour used in drop-size measurements by means of tubes of known diameter. (Data from measurements made at Philadelphia Quartermaster Depot and from* Transactions, American Geophysical Union, *24th Annual Meeting, April 23–24, 1943.)*

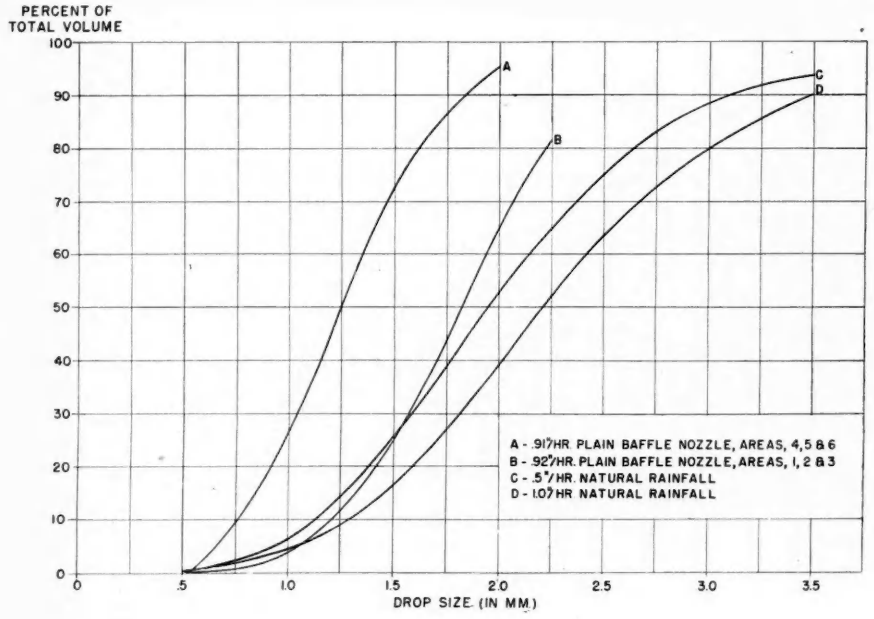


FIG. 9. *Comparison of cumulative volume curves for natural and simulated rainfall. (Natural rainfall data obtained from* Transactions, American Geophysical Union, *24th Annual Meeting, April 23–24, 1943*— Part II.)

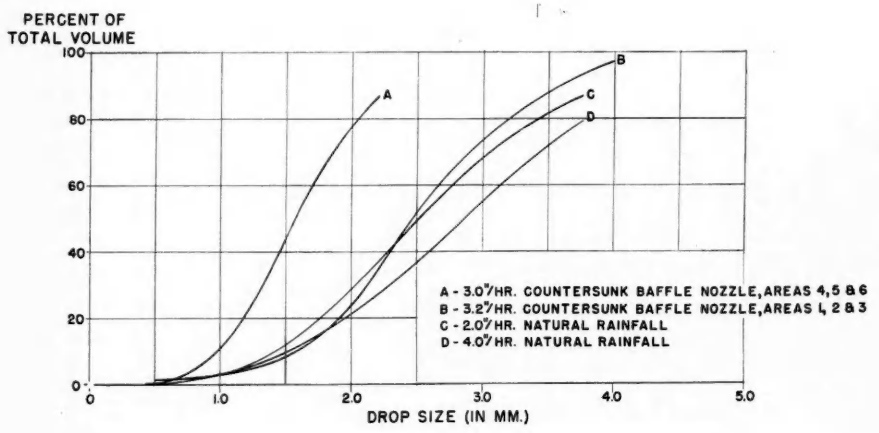


FIG. 10. *Comparison of cumulative volume curves for natural and simulated rainfall. (Natural rainfall data obtained from* Transactions, American Geophysical Union, *24th Annual Meeting, April 23–24, 1943*— Part II.)

To ascertain whether the three nozzles developed furnished drop sizes similar to those found in natural rainfall of 1 inch, 3 inches, and 0.1 inch per hour, cumulative volume curves for each nozzle were drawn using the data obtained from the measurements of artificial rainfall, and these were compared with the cumulative volume curves of natural rainfall of corresponding intensities reported in the work of Parsons and Laws [4].

For corresponding intensities, larger drops occur in natural rainfall than in the simulated rainfall. The diameter of the median * drop size, however, for the three intensities created was of the same order as that found in natural rainfall (Table IV and Figures 9–11).

TABLE IV

Nozzle	Average intensity (in./hr.) Simulated rainfall	Natural rainfall	Diameter of median drop (mm.) Natural rainfall	Simulated rainfall (B)	(A)
Flat baffle	0.92	1	2.2	1.9	1.25
Countersunk baffle	3.07	3	2.75	2.5	1.58
Rotary sprinkler	0.114	0.1	1.45	1.4	1.38

The slope of the cumulative curves (Figures 9–11) at the point of reversal from convex to concave indicates the relative dispersion of the drop sizes in each area. From the curves it is seen that drop-size dispersions in natural rainfall exceed the dis-

* The median drop size is the drop-diameter interval which divides the total volume into two equal parts. It is determined by the intersection of the 50-percent volume abscissa and the cumulative volume curve.

persions in artificial rainfall. Relative displacement of the cumulative curves along the drop-diameter axis (abscissa) provides corresponding information concerning the displacement of drop-size distributions. As noted in the computation of median drop sizes, the distribution displacement along the abscissa of artificial rainfall in the selected areas and that of natural rainfall of corresponding intensity are of the same order.

In view of the fact that in natural rainfall Parsons and Laws [4] found that ". . . rainfall samples of nearly equal intensity displayed wide differences in (drop-size) distribution," the results obtained with the three nozzles appear to be acceptable for testing purposes. The values obtained by Parsons and Laws for the median drop sizes for natural rainfall shown in Table IV are averages of ten observations. Under these conditions it is not improbable that at least one or more of the natural-rainfall measurements compared more closely to the measurements obtained from the nozzles described herein.

Velocity Measurements

To this point in the tests, the heights at which the nozzles had been placed were based on the values cited in the literature as necessary for drops to attain terminal velocity. The velocity of a single drop 2.5 mm. in diameter falling from various heights was determined experimentally to permit comparisons with the measurements reported by other investigators. The velocity measurements were made by means of stroboscopic photography, using a flashing rate of 3,000 times per minute for the drop falling from heights of 3, 6, and 9 feet, respectively, and 6,000 times per minute for the drop falling from heights of 12, 15, 18, and 21 feet, respectively. The height of fall of the drop was then plotted against its velocity (Figure 12).

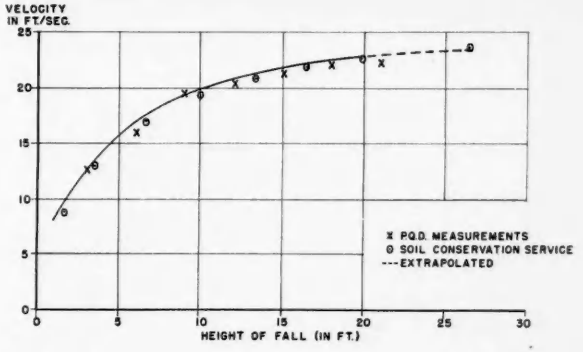


FIG. 12. *Fall velocity of a 2.5-mm. water drop from various heights. (Data from measurements made at Philadelphia Quartermaster Depot and from* Transactions, American Geophysical Union, *24th Annual Meeting, April 23–24, 1943.)*

The comparative values for a similar-size drop obtained by Laws [5] are also shown. Good agreement exists between the measured and the reported velocities in the range of from 3 to 21 feet. Since at the Depot the velocity measurements were made

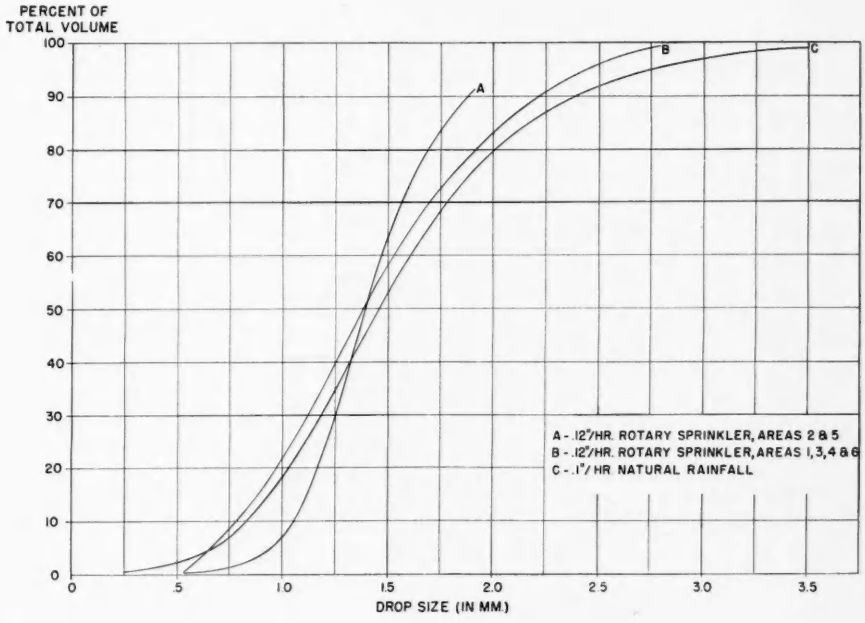


FIG. 11. *Comparison of cumulative volume curves for natural and simulated rainfall. (Natural rainfall data obtained from* Transactions, American Geophysical Union, *24th Annual Meeting, April 23–24, 1943—* Part II.)

only to 21 feet, the curve was extrapolated to include the height of 26.3 feet at which the velocity of a 2.5-mm. drop was measured by Laws [5]. Difficulty was encountered in determining the point at which terminal velocity was reached, as there was very little difference between readings of velocity above heights of 18 feet. The height from which a drop of 2.5-mm. diameter must fall in order to attain 95 percent of its terminal velocity was found to be 18.2 feet. Direct interpolation of the values reported by Laws [5] for a 2-mm. and 3-mm. drop provided a value of 20 feet as the height required for a drop of 2.5 mm. to attain 95 percent of terminal velocity. On the basis of the check measurement and the values reported in the literature, the heights shown in Table V were used as a guide for establishing the minimum nozzle elevation necessary to insure that the falling drops have attained 95 percent of their terminal velocity upon striking the subject. Approximately 10 feet were allowed for subject and obstacle-course height.

TABLE V

Size of drop (mm.)	Height to attain 95% terminal velocity (ft.)	Size of median drop from nozzles (mm.)	Height to attain 95% terminal velocity (interpolated value) (ft.)
1	7.2	1.45	11.0
2	16.4	1.9	15.6
3	23.6	2.7	21.2

Other factors which are known to affect drop sizes, intensities, and velocities of rainfall include wind and water and air temperature. With the exception of wind, these factors were considered as relatively non-important in this study. The use of an indoor site permitted the elimination of the wind factor. The temperature of both the water and the surrounding air affects the drop sizes and intensities through changes in surface tension of the water, with subsequent effects on the breaking up of the water stream into drops. However, it was found that very large temperature differentials must occur before the thermal effects cause significant differences in test results.

Conclusions

The nozzles developed, although not perfect in their reproductions of actual weather conditions, furnish simulated rainfall with respect to the three most important features, intensity, drop sizes, and velocity having the same order of magnitude as those found in natural rainfall.

Differences known to exist between areas were not considered critical in view of the fact that a relatively large test site was still available in which controlled tests could be conducted.

Acknowledgments

Acknowledgment is made of the assistance of the following individuals and agencies; their cooperation greatly facilitated the completion of the work on this project in a shorter time than otherwise would have been possible:

Philadelphia Quartermaster Depot—Capt. Stanley Backer, Officer in Charge of the Research Laboratory Section, under whose direction the original report and this publication were prepared, for his cooperation, constructive criticisms, and comments.

Frankford Arsenal—Maj. Culbertson, Dr. Myers, and Mr. E. Thilo, for their work of photographing a falling waterdrop.

Philadelphia Navy Yard—Comdr. Watson, for furnishing personnel to assist in machining the baffle-type nozzles.

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Active Chlorine Compounds—Their Chemistry and Application

Albert G. Chenicek*

Active chlorine compounds form a group of substances capable of many useful reactions. In addition to their well-known bleaching properties, newer compounds of this class are finding unusual and specialized applications in the textile industry. It would appear that if the nature of these compounds were better known they would find wider usage in the industry. This article describes the chemistry of some of the known active chlorine compounds.

THE group of substances designated as active chlorine compounds is characterized by the presence in the molecule of one or more chlorine atoms with a unit positive charge. The chlorine atoms are usually attached to oxygen, nitrogen, or sulfur atoms. Both inorganic and organic compounds are represented [4].

Such compounds are known as "active" chlorine compounds, because of their pronounced oxidizing power and general reactivity. The unit positive chlorine atom distinguishes these substances from other compounds also having oxidizing powers, but which contain chlorine atoms with a positive charge of three, five, or seven. These chlorine atoms occur in compounds such as chlorous acid, $HClO_2$; chloric acid, $HClO_3$; and perchloric acid, $HClO_4$. The typical reactions of active chlorine compounds are not characteristic of these compounds.

The reactions of active chlorine compounds are, in general, the same as those of the element chlorine—addition to double bonds, oxidation, chlorination, and hydrolysis to form hypochlorous acid. All active chlorine compounds react with chloride ions to form chlorine. This method often is used to determine the amount of "available" chlorine contained in an active chlorine compound.

The usage of the terms "active chlorine" and "available chlorine" has been the source of some confusion, particularly since there have been few published attempts to standardize terminology. An article [*20*] written in 1920 attempts to explain the source of the oxidizing power of bleaching powder based upon the reaction of chlorine with lime. No definitions are given and the terms "active" and "available" are considered to be synonomous. A later article [19] proposes the following definition: "Available chlorine is the weight of chlorine given by reacting a given weight of sample under ideal conditions with an excess of HCl, the results to be expressed as percent." Such a definition includes compounds other than those containing unit positive chlorine atoms such as sodium chlorite, which reacts with hydrochloric acid to form chlorine. Active chlorine is not defined.

Present accepted usage is that active chlorine is the percent by weight of positive chlorine in the molecule and is applied to organic compounds. Available chlorine is twice the value of active chlorine and is used for inorganic compounds. The terminology in this article conforms to this usage. It would be preferable to have all calculations of oxidizing power on the basis of active chlorine contents, but convention dictates otherwise. These figures are given in Table I as a means of comparing organic and inorganic compounds.

The calculation of active and available chlorine contents is best illustrated by an example. Sodium hypochlorite, NaOCl, contains 47.6 percent chlorine. Since the chlorine atom is positive, the oxidizing power corresponds to that of one molecule of chlorine. The percent of active chlorine, therefore, is 47.6 percent, while the available chlorine content is 95.3 percent. In contrast, chloroform, $CHCl_3$, which contains 89.1 percent chlorine, has 0 percent of active chlorine because none of the chlorine atoms is positive.

Chlorine

Elementary chlorine is the raw material for all active chlorine compounds. It normally is prepared

* Interchemical Corporation Research Laboratories.

by the electrolysis of brine obtained by pumping water into deposits of salt. The by-product of the electrolysis of brine is generally impure sodium hydroxide solution, although some processes give metallic sodium, sodium nitrate, or sodium carbonate. The magnitude of the chlorine industry is indicated by the production for 1944, when, exclusive of Government plants, 1,252,768 tons of chlorine were produced.

In order to ship economically as a liquid, chlorine must be condensed by cooling and compression after it is removed in the gaseous state from the electrolytic cells. Since the gas is diluted with non-condensable gases such as nitrogen, all the chlorine cannot be liquefied economically. However, this residual diluted chlorine is used for the preparation of sodium hypochlorite, bleaching powder, and sulfur chlorides.

Hypochlorites

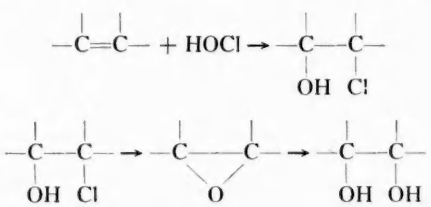
The best-known solid active chlorine compound is bleaching powder, $CaOCl_2$, which is made by passing chlorine into calcium hydroxide and water [21]. This substance has only one positive chlorine atom, and since the other chlorine atom is negative, the available chlorine content of the molecule is 55.9 percent. It is sold, however, in an impure form containing 35–37 percent available chlorine. Calcium hypochlorite, $Ca(OCl)_2$, commonly confused with ordinary bleaching powder, has two positive chlorine atoms, and an available chlorine content of 99.3 percent. Again, the commercial product has a lower available chlorine content—70 percent. The true hypochlorite is a much more efficient bleaching and sterilizing agent than bleaching powder because of the difference in available chlorine content. One of the most recent uses for such hypochlorites was for shrinkproofing woolen Army socks [30]. The tremendous volume of production of these two compounds is revealed by the figures for 1944. In that year, about 24,000,000 tons of bleaching powder, and about 7,000,000 tons of calcium hypochlorite were produced.

Sodium hypochlorite is also a widely used active chlorine compound. It is not isolated as a solid, but is sold in solution, or prepared from caustic soda solution and chlorine just before it is needed for use. It may be made also from bleaching powder and sodium carbonate or bicarbonate. It is particularly useful for bleaching fine cotton goods. Dakin's solution, first used as an antiseptic and sterilizing agent for wounds in the first World War, is a sodium hypochlorite solution. Because it does not materially damage living cells or lose its potency in the presence of blood serum, it is sometimes employed in preference to iodine or carbolic acid. Dakin believed that the tissue proteins were first converted into chloroamines, and that these slowly liberated active chlorine. Javelle water, first prepared in 1785, is made by passing chlorine into a solution of sodium or potassium carbonate. This solution is probably mostly hypochlorous acid, with some sodium or potassium hypochlorite.

All these compounds are salts of hypochlorous acid, HOCl, which exists only in solution. This acid is weaker than carbonic acid and so may be liberated from its salts by treatment with carbon dioxide—a convenient method of preparing solutions of the acid. It may also be formed by passing chlorine into water.

On the basis of its active chlorine content, hypochlorous acid should be a more efficient bleaching agent than sodium hypochlorite. However, other factors such as pH and stability affect the action of active chlorine compounds, so that their oxidizing power is not a true measure of their efficiency.

The addition of hypochlorous acid to organic compounds with double bonds produces chlorohydrins, which can be converted to the corresponding oxide and then to the glycol, or to the glycol directly [15].



These reactions are the foundation upon which a large synthetic chemical industry has been built in the last decade. In the newer processes, however, the olefin oxide is produced directly by oxidation.

There are other ways in which hypochlorous acid, and therefore hypochlorites, may be prepared. These methods are based upon forming active chlorine compounds which hydrolyze to produce hypochlorous acid. Chlorine monoxide, Cl_2O, is one such compound [21]. It is a gas which reacts with water to form two moles of hypochlorous acid. By this means, hypochlorites such as calcium hypochlorite can be prepared which are practically free of chlorides. Such compounds cannot be prepared from chlorine without extensive purification. Chlorine monoxide can be prepared by the action of chlorine

upon compounds such as mercuric oxide or sodium carbonate in the presence of small amounts of water. Chlorine monoxide is a strong oxidizing agent and a powerful chlorinating compound; it adds to double bonds to form chloroalkyl hypochlorites.

Certain organic compounds also form hypochlorite salts. These are quaternary ammonium hydroxides, which are strong bases and form salts of the type R_4NOCl [22].

Another kind of organic hypochlorite has the general formula ROCl, where *R* may be one of a wide variety of alkyl groups and some aryl groups. Aliphatic hypochlorites are volatile yellow liquids with an irritating odor. They may be prepared by the action of hypochlorous acid or its salts upon an alcohol [27, 28]. The action of excess hypochlorite at elevated temperatures results in the formation of chloroform. Aromatic hypochlorites are prepared from the corresponding phenol.

The primary and secondary alkyl hypochlorites are very unstable, whereas the tertiary compounds are quite stable. Aromatic hypochlorites are very unstable and rearrange to form nuclear substituted chlorine compounds if all the positions in the ring are not already substituted.

Decomposition of primary aliphatic hypochlorites results in the formation of a complex mixture of oxidation and chlorination products. Thus ethyl hypochlorite gives acetaldehyde, ethyl acetate and chloroacetates, ethyl alcohol, ethyl acetal and chloroacetals, hydrochloric acid, and chlorine.

One of the most interesting reactions of alkyl hypochlorites is their addition to double bonds, which is illustrated by the preparation of beta-monochloroethyl ether from ethyl hypochlorite and ethylene.

$$H_3C{-}CH_2OCl + H_2C{=}CH_2 \rightarrow H_3C{-}CH_2{-}O{-}CH_2CH_2Cl$$

In general, the unsaturated compound and the chlorine are led separately into an alkaline alcohol solution, the alkali being present in slight excess of that required to neutralize the hydrochloric acid which is formed. There is produced an alkyl hypochlorite which reacts with the olefin to form a chloroether.

Amyl hypochlorite has been patented as a shrinkproofing agent for wool.

Thiohypochlorites (RSCl) are also known although very little study has been made of their reactions. The compounds are probably more reactive and less stable than the normal hypochlorites. One common reaction in which a thiohypochlorite may be formed as an intermediate occurs in the preparation of beta-dichloroethyl sulfide, or mustard gas, from ethylene and sulfur dichloride.

$$SCl_2 + 2H_2C{=}CH_2 \rightarrow ClCH_2{-}CH_2{-}S{-}CH_2CH_2Cl$$

Sulfur dichloride, as shown by the above reaction, reacts with olefins in the same manner as chlorine monoxide. Sulfur monochloride, S_2Cl_2, has no known oxygen analog. Its reactions are those of the dichloride.

N-Chloroamines

There are three chlorine compounds of nitrogen: chloroamine, NH_2Cl; dichloroamine, $NHCl_2$; and trichloroamine, NCl_3, better known as nitrogen chloride.

Chloroamine may be prepared by the action of chlorine or hypochlorites on ammonia. It is a very unstable yellow oil, but its aqueous solutions may be stabilized by the addition of small amounts of ammonia. Chloroamine probably is formed during the process of water purification, in which both chlorine and ammonia are used. It is an effective sterilizing agent.

Dichloroamine is also an oil. Very little else is known of this substance because it is difficult to isolate. This may be due to its disproportionation into chloroamine and nitrogen trichloride.

Nitrogen trichloride has an available chlorine content of 176.8 percent. This figure is a good measure of its tremendous oxidizing power, which is 1.77 times that of chlorine. Very great care must be used in handling this compound because it reacts explosively with many organic compounds. For example, contact with stopcock grease should be avoided in laboratory setups. Nitrogen trichloride is usually prepared by the action of excess hypochlorite or chlorine on ammonia or its salts.

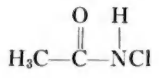
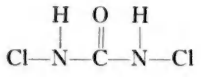
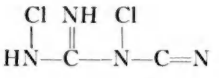
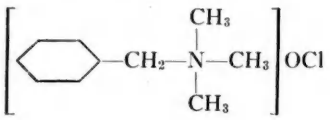
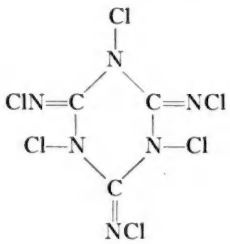
The addition of chloroamine and nitrogen trichloride to a wide variety of unsaturated organic compounds results in the formation of the corresponding amines and *N*-dichloroamines [7, 8, 9, 11, 12, 13, 14].

$$NCl_3 + H_2C{=}CH_2 \rightarrow ClCH_2{-}CH_2NCl_2$$

Their reactions with Grignard reagents [5, 10] and zinc alkyls [6] have also been studied.

N-chloroalkyl and aryl amines can be prepared by the same reactions as those by which the chloroamines are formed. An exception is the preparation

TABLE I. SOME ACTIVE CHLORINE COMPOUNDS WITH CONSTITUTION AND APPLICATIONS

Inorganic Compound	*Formula*	*Chlorine Content* % *Active*	% *Available*	*Uses*
Bleaching powder	$CaOCl_2$	27.95	55.9	Bleaching, sterilization
Calcium hypochlorite	$Ca(OCl)_2$	49.6	99.2	Bleaching, sterilization
Sodium hypochlorite	NaOCl	47.6	95.3	Bleaching, sterilization
Hypochlorous acid	HOCl	67.6	135.2	Synthesis: chlorohydrins, glycols
Chlorine monoxide	Cl_2O	81.6	163.2	Preparation of hypochlorites
Sulfur dichloride	SCl_2	68.9	137.8	Chlorination of ores, hydrocarbons, fatty acids; preparation of insecticides, dyes
Monochloroamine	H_2NCl	68.9	137.8	Sterilization, water purification
Dichloroamine	$HNCl_2$	82.5	165.0	
Nitrogen trichloride	NCl_3	88.4	176.8	
Organic Compound				
Ethyl hypochlorite	$H_3C\text{-}CH_2\text{-}OCl$	44.1		Preparation of chloroalkyl ethers
Chloroethyl hypochlorite	$ClCH_2\text{-}CH_2\text{-}OCl$	61.7		Preparation of dichloroethyl ether
N-Chloroacetamide		38.0		
N-Dichlorourea		55.0		Bleaching, chemical syntheses
N-Dichlorodicyandiamide		46.4		Sterilization, antiseptic, germicide
Benzyl trimethyl ammonium hypochlorite		18.5		Sterilizing, antiseptic, germicidal agent
N-Hexachloromelamine		63.9		Impregnation of bandages, sheets, etc.; antiseptic

of methylene chloroamine by the action of monochloroamine on formaldehyde.

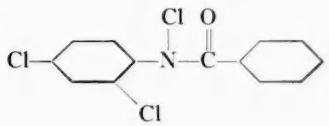
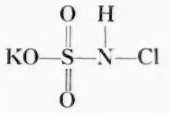
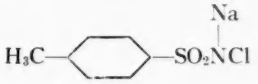
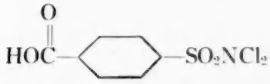
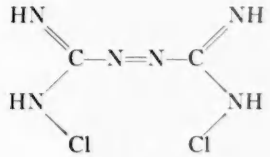
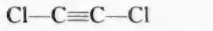
The lower aliphatic *N*-chloroamines are all unstable yellow oils. The aromatic chloroamines, like the aromatic hypochlorites readily rearrange to form nuclear substituted compounds [18].

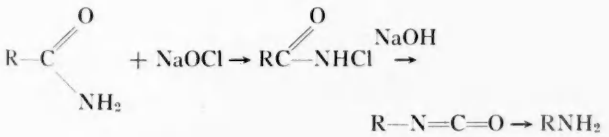
N-Chloroamides

Acid amides also react with hypochlorites, hypochlorous acid, and chlorine to form *N*-chloro- derivatives. The mono-chloro- compounds are colorless, crystalline, and fairly stable. The dichloro- derivatives are extremely unstable, corrosive, yellow liquids whose stability increases with increasing molecular weight. In general, the *N*-monochloroamides are formed by chlorination and the *N*-dichloro- compounds by the reaction of the amide with hypochlorous acid.

Treatment of acid amides with sodium hypochlorite results in the formation of an *N*-chloroamide. In the presence of excess alkali this compound undergoes the well-known Hofmann degradation to form a primary amine [17].

TABLE I.—(*Continued*)

Organic Compound	*Formula*	*Chlorine Content* % *Active*	% *Available*	*Uses*
"Impregnite"		35.5		Protection against mustard gas
Potassium chloroamido-sulfonate		20.9		
"Chloramine T"		18.1		Sterilization, antiseptic, germicide
"Dichloramine T"		34.1		Sterilization, antiseptic, germicide
"Halozone"		29.8		Water sterilization
"Azochloramide"		38.8		Antiseptic, germicide
Dichloroacetylene		74.7		



Urea, which is the diamide of carbonic acid, forms monochloro- and dichloro- derivatives [1, 2, 3]. The presence of alkaline reagents is necessary to obtain good yields. Reaction with sodium hypochlorite, especially at elevated temperatures, causes decomposition to carbon dioxide and nitrogen. The amount of nitrogen obtained is always less, by a constant factor, than the theoretical value. This is the general method used for the quantitative determination of urea.

Bleaching of cloth containing urea resins often causes tendering. This may be due to the formation of *N*-chloro- compounds which may oxidize the cellulose as well as decompose later to form hydrochloric acid, which attacks the cellulose. The result in both cases is a weakening of the fibers.

Compounds related to urea, such as biuret, carbamates, guanidine, dicyanodiamide, cyanuric acid, ammelide, ammeline, and melamine all form active chlorine compounds. These derivatives of the last five compounds exhibit a stability which is considerably better than that of other *N*-chloro- acid amides [23, 24, 25, 26]. Chlorinated melamine is of particular interest. A hexachloro- compound can be prepared which contains 64 percent active chlorine. It is a crystalline solid, soluble in many organic solvents, but forming stable solutions only in chlorine containing solvents such as carbon tetrachloride. Solutions and slurries of the members of this group are of value in impregnating fibrous materials to impart antiseptic properties to them [26].

Another compound, used for the impregnation of clothing to provide protection against mustard gas,

is noted by Fieser [16]. This is the British "Impregnite," 2,4-$Cl_2C_6H_3N(Cl)COC_6H_5$. It reacts with mustard gas to give oxidized and chlorinated products which are no longer vesicants.

N-Chloro-Sulfonamides

Sulfonamides are also capable of forming active chlorine compounds. Very few inorganic derivatives have been described. Sulfamide, $H_2NSO_2NH_2$, reacts with bleaching powder to form sulfone-chloroamide salts. Potassium chloroamidosulfonate, KSO_3NHCl, has been prepared by treating the corresponding amide with hypochlorous acid in the cold.

While aliphatic sulfonamides do not form active chlorine compounds very readily, the aromatic sulfonamides do, and these constitute a group of very useful substances. The best-known member of the group is "Chloramine T," which is the sodium salt of *N*-chloro-para-toluene sulfonamide. This compound has found wide use as a sterilizing agent because it is stable and water-soluble. "Dichloramine T," or *N*-dichloro-para-toluene sulfonamide, is not water-soluble. It has been used as a solution in a chlorinated paraffin called "Chlorcosane." The corresponding *N*-chlorobenzene sulfonamides also have been prepared.

Another derivative is "Halozone," which is *p*-$HOOC\text{-}C_6H_4SO_2NCl_2$. This is pelleted with sodium carbonate, and when a pellet is added to water a soluble sodium salt is formed. The tablets are used for the sterilization of drinking water. This is the tablet which was used successfully by the Armed Forces during the war.

Amino Acids and Proteins

Proteins and their component amino acids possess amide and amine groups which should make them convertible to active chlorine compounds. Chlorocasein, chlorofibrin, chlorogluten, and chloro-ovoprotein have been obtained [29]. They are light-yellow powders of irritating odor, are rather hygroscopic, and are partly soluble in cold water. No evidence has been presented that these derivatives are active chlorine compounds, although it is very likely that they are.

If Dakin's concept of the action of sodium hypochlorite as a sterilizing agent is correct, products of this type are formed upon the application of hypochlorites to tissue. The lower solubility and decreased rate of hydrolysis of the *N*-chloroamino acid should result in a prolonged antiseptic action. The greater effectiveness of other *N*-chloro- compounds as sterilizing agents may be due to these same properties.

Other Active Chlorine Compounds

"Azochloramide," or *N.N'*-dichloroazodicarboxamidine, is sold as a sterilizing agent, mixed, usually, with enough sodium chloride to form an isotonic solution in water. It is prepared by the action of sodium hypochlorite upon a guanidine salt.

Calcium cyanamide gives a very explosive substance on treatment with chlorine. This may be the compound $Cl_2N\text{-}CN$.

Carbonyl hypochlorites, of the general formula $RCO(OCl)$, have been shown to exist by their addition to olefins to form chloroalkyl esters.

Compounds which fall within the scope of the definition of active chlorine compounds are the chloroacetylenes. These have the chlorine atoms attached to carbon atoms. Acetylene, C_2H_2, forms both mono- and di-chloro- compounds of formulas HC_2Cl and C_2Cl_2. Both of these substances hydrolyze to give hypochlorous acid. They are highly explosive when exposed to air and must be handled with extreme care. They can be prepared by the action of hypochlorous acid solution upon calcium carbide.

There are other known active chlorine compounds, but they fall in the general classifications already discussed. In addition, some compounds such as phosgene and sulfuryl chloride are on the borderline between active and inactive chlorine compounds in that under certain conditions they exhibit some of the characteristic reactions of active chlorine compounds, but in general behave as substances containing negative chlorine atoms.

Summary

Certain inorganic active chlorine compounds are widely used in the fields of sterilization, bleaching, and chemical synthesis. They have also been employed in the process used by the Army for shrinkproofing of socks. Their worth has been proved and they will continue to be used because of their low cost and ease of preparation.

Organic active chlorine compounds, in contrast, have been rather limited in number and applications. Their higher cost will limit their use except as special antiseptic and sterilizing agents and possibly in certain chemical syntheses. Their potentialities along these lines are great and probably have hardly been

touched in terms of the number of products which can be made.

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ABSTRACTS

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A list of periodicals, the abbreviations used in references to articles, and the addresses of publishers of English language publications is given on page 84 of the February, 1946, issue.

ANALYSIS: TESTING: LABORATORY METHODS

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Analysis of Cellulose

Oxy- and hydrocellulose. III. Determination of the carbonyl groups in hydrocelluloses. Ernst Geiger and Alfred Wissler. *Helv. Chim. Acta.* **28**, 1638–47 (1945) (in German); cf. *C.A.* **40**, 1031[2] (through *Chem. Abstr.* **40**, 1313[1] (Mar. 10, 1946)).

By condensing the hemiacetal (potential) aldehyde group of a hydrocellulose (*I*) with primary amines, hydrazines, or hydrazides, it was possible to introduce either acid or basic groups into the molecule. By ion-exchange, these groups serve to fix dyestuff ions on *I*, and these can be split off subsequently and determined colorimetrically. Each potential aldehyde group is equivalent to 1 dyestuff ion, and thus furnishes a quantitative measure of the number of such groups present per g. of *I*, and may be used in determining the molecular weight of *I*. Values obtained in this way are in harmony with those found by the use of osmotic pressure, the ultracentrifuge, and by viscosity measurements. On acid hydrolysis, each fragment appears to retain 1 aldehyde (hemiacetal) group. The greater part of the article reviews the other methods used in determining CO_2H and CHO groups and the degree of polymerization of *I*. The mathematical basis for G. and W.'s methods is also discussed. In determining the CO_2H (and(or) SO_3H) groups, a very slight modification of Weber's reversible methylene blue number was used (cf. *C.A.* **36**, 653[5]). The (potential) aldehyde groups in *I* were determined as follows: 0.1–0.2 g. *I* was treated at 20–30° for 2–3 days with a 5% solution of acethydrazide pyridinium chloride or of trimethylacethydrazide ammonium chloride in a borate buffer solution (pH 8.5). *I* was then filtered through a small, fritted-glass filter tube, and washed and macerated with successive 10–15-ml. portions of picric acid solution (0.4 g. per l.) (*II*). In each case the solution was drawn off, and a total of 100 ml. *II* was used in the treatments. The colored residue was removed from the tube, pressed between filter papers, and transferred to another filter tube, where it was decolorized by stirring with successive 10–15-ml. portions of 0.01 *N* NaOH (with a total of 80 ml. of this solution) and filtering quantitatively after each treatment. The combined alkaline picrate solutions were then acidified with 10 ml. of 0.1 *N* HCl, made up to 100 ml., and the picric acid content determined colorimetrically. A factor permits the conversion of the percentage of picric acid into the number of CHO groups per g. *I*. 22 references.

Text. Research J. May 1946

Color-Fastness Tests

Color-fastness tests. A. R. Docking. *Text. J. Australia* **20**, 241–4 (1945) (through *Chem. Abstr.* 40, 1039[8] (Feb. 20, 1946)).

Tests are described for measuring the effects of the weather, laundering, and wear. 17 references.

Text. Research J. May 1946

Control of Bleaching Process

Determination of residual lignin. A colorimetric method for the control of the bleaching process. Hans W. Giertz. *Svensk Papperstidn.* **48**, 485–9 (1945) (German and English summaries) (through *Chem. Abstr.* **40**, 1315[3] (Mar. 10, 1946)).

The amount of residual lignin in bleached pulp is determined by dissolving the pulp in 76% H_2SO_4. The pulp is first extracted with benzene-alcohol; 1 g. of extracted pulp (or 0.5 g. of partially bleached pulp) is dissolved in 70 cc. H_2SO_4

at 25°. To prevent the formation of gelatinous lumps, the flask is evacuated. The mixture is then shaken to complete the solution and deaerated by placing it under a vacuum for 10 min. The solution is filtered through a Gl Jena glass filter and the brown color of the filtrate is measured colorimetrically, with a Hellige-type photocell colorimeter. The light absorption, log $I_0/I + \epsilon dc$, in which ϵ is the extinction coefficient, d the thickness of the layer, and c the concentration, is designated as the residual lignin number. The colorimetric determination is claimed to be a suitable method for the control of the bleaching operation. A definite relation exists between the brightness of the pulp and the residual lignin number.
Text. Research J. May 1946

Determination of Xylose

The quantitative estimation of xylose. L. J. Breddy and J. K. N. Jones. *J. Chem. Soc.* **1945**, 738–9 (Nov. 1945).

Xylose condenses with benzaldehyde dissolved in methyl alcoholic hydrogen chloride forming the sparingly soluble dibenzylidene dimethyl acetal of xylose. A quantitative method of estimation of the sugar has been developed, based on the formation of this derivative.
Text. Research J. May 1946 Authors

Effect of Low Temperature on Rubber

A new method for determining the resistance of soft-rubber products to low temperatures. J. Aengeneyndt and W. Kesternich. *Rubber Chem. Tech.* **18**, 401–4 (Apr. 1945).

The method involves the measurement of the deflection of a strip of material supported at each end when a load is applied at the center. The deflection is measured as a function of time and temperature. Using this method, it is simple to determine the lowest temperature at which the material is still completely elastic as well as the hardening point. W. E. Davis
Text. Research J. May 1946

Electron Microscopy

Surface replicas for electron microscopy. Lars Thomassen, Robley C. Williams, and Ralph W. G. Wyckoff. *Rev. Scientific Instruments* **16**, 155–6 (June 1945).

An improved technique for obtaining surface replicas of specimens for examination with the electron microscope is described. Formerly, it was customary to prepare a direct replica film of formvar or to make a silica film from a preliminary polystyrene imprint by vacuum deposition. The authors have found that electron micrographs of improved contrast and sharpness may be obtained by shadow-casting a film of chromium on a formvar replica. The chromium is obliquely evaporated onto the surface of the formvar replica so that variations in electron transparency or "shadows" are formed. T. J. Dietz
Text. Research J. May 1946

A shadow-casting adaptor for the electron microscope. Thomas F. Anderson. *Rev. Scientific Instruments* **17**, 71–2 (Feb. 1946).

An apparatus is described for shadow-casting metallic coatings directly onto a specimen which is mounted in the electron microscope in position for viewing. The author finds that this technique not only avoids the damage that may result from handling during evaporation but also permits observation of the specimen during the process so that the deposition may be halted when optimum contrast is obtained.
Text. Research J. May 1946 T. J. Dietz

Polarographic Analysis

The Polarographs. S. Matthews. *J. Soc. Dyers and Colourists* **61**, 164–5 (July 1945).

The Polarograph is a tool for the analysis of solutions which depends upon the reduction or oxidation of dissolved substances at a dropping mercury electrode, provides a means of applying a known variable potential between the solution and the dropping mercury, and includes a galvanometer for measuring the current. It may be used for quick and accurate qualitative and quantitative analysis of (especially) metal ions in solution and many dyestuffs. K. S. Campbell
Text. Research J. May 1946

Stiffness of Rubber

The stiffness of rubber on bending, or its dynamic hardness. The method of resonance vibrations. K. H. Reiss. *Rubber Chem. Tech.* **18**, 394–400 (Apr. 1945).

The method of making the measurements is described briefly. Results for Buna-S and natural rubber with various fillers are presented in tabular form and also as graphs of dynamic bending modulus vs. hardness. The relationship between these 2 variables was found to depend on the character of the filler. W. E. Davis
Text. Research J. May 1946

Tear-Resistance

The evaluation of tear-resistance in elastomers. F. L. Graves. *Rubber Chem. Tech.* **18**, 414–23 (Apr. 1945).

The effect of specimen shape on the proportion of applied stress actually used in tearing the sample is discussed. It is shown that this proportion is small for the usual crescent-shaped test piece, and a new angle-shape, which does not require razor-nicking before testing, is described. Data for Hevea, GR-S, neoprene, and hycar are presented to show that much more uniform results are obtained with the new shape of specimen.
Text. Research J. May 1946 W. E. Davis

Viscosity of Cellulose

Macromolecular compounds. CCCXVII. Cellulose. 88. Determination of the viscosity number of cellulose in Schweizer reagent by measurements at higher concentrations. E. Husemann and G. V. Schulz. *J. makromol. Chem.* **1**, 197–202 (through *Chem. Abstr.* **40**, 201[5] (Jan. 10, 1946)).

The viscosity number (the specific viscosity at 0 concentration) can be derived for cellulose dissolved in cuprammonium hydroxide from measurements at higher concentra-

tions by an equation suggested by H. and S. (*C.A.* **37**, 5861[6]) for cellulose nitrates in acetone. With a value of 0.28 for the constant in this equation, the viscosity numbers were calculated from the specific viscosity data obtained at varying concentrations. The plot of specific viscosities against specific viscosity concentration gives straight lines. The equation can be used for concentrations corresponding up to a specific viscosity of at least 2.

Text. Research J. May 1946

Degradation of Wool

Bacterial degradation of woolen felts. C. Binns. *Proc. Tech. Sect., Paper Makers' Assoc. Gt. Brit. & Ireland* **25**, 280–5 (1944) (through *Chem. Abstr.* **40**, 206[3] (Jan. 10, 1946)).

The extent of bacterial degradation of wool has been assessed by the loss of tensile strength of woolen yarn under incubation. The attack by bacteria is dependent on the source of the water used in *paper manufacture* and is generally increased by the presence of phosphates. Certain methods of felt treatment (details not disclosed) offer a high order of protection. The intermittent treatment of felts with bactericidal substances (*e.g.*, Cl, $HgCl_2$, $CuSO_4$, phenol, and chlorinated derivatives) will probably be effective only if used in relatively large concentrations. Immunity from attack is favored by acid conditions.

Text. Research J. May 1946

CHEMICAL AND PHYSICAL RESEARCH

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Bleaching Linters

Bleaching of cellulose in linters. L. Thoria. *J. Indian Chem. Soc., Ind. & News Ed.* **7**, 123–32 (1944) (through *Chem. Abstr.* **40**, 203[8] (Jan. 10, 1946)).

Indian linters, mechanically cleaned, were kier-boiled with 6% NaOH (*I*), 8% NaOH (*II*), and 7% NaOH and 1% Na aluminate (*III*) (each with 1% Igepon T); *II* gave the best results. *I*, bleached at pH 8.5, showed the maximum whiteness with minimum degradation and lowest Cl consumption; the best results were obtained with 8–15% HOCl. Under these conditions, $NaHCO_3$ exerts a specific and advantageous influence on the bleaching process; no explanation is offered for this action. The optimum bleaching time is about 1.5 hrs.; beyond this there is a rapid degradation of linters. Experiments with *III* showed that the optimum concentration of available Cl was about 1.5 g./l.; any excess above this amount leads almost exclusively to degradation of the cellulose. The results show the need of the joint investigation of kier-boiling and bleaching.

Text. Research J. May 1946

Cellulose Ester and Ether Films

The relaxation process in films of cellulose ethers and esters. P. V. Kozlov. *Trudy Konferentsii Vysokomolekulyar. Soedineniyam, Akad. Nauk S.S.S.R., Otdel. Khim. Nauk i Otdel. Fiz.-Mat. Nauk* **1**, 12–14 (1943) (pub. 1945); cf. *C.A.* **37**, 3268[5] (through *Chem. Abstr.* **40**, 266[1] (Jan. 20, 1946)).

Many anomalous effects in the stretching and relaxing of films of cellulose ethers and esters prepared in the usual way are due to internal strains set up between the surface layers of the films that are oriented by the surface on which they form, and the inner layers of the film that are differently oriented. Thus anomalies of optical anisotropy are not due to processes resembling crystallization. The anomalous effects can be avoided by forming the films on a Hg surface.

Text. Research J. May 1946

Cellulose Xanthate

Dispersion of cellulose by cellulose xanthate solutions. Philip C. Scherer. *Rayon Text. Mo.* **27**, 22–6 (Jan. 1946).

While the results obtained varied rather more widely than desirable, the general indications from the experiments were that cellulose, to a degree, may be dispersed by the action of solutions of purified cellulose xanthate. In the case of the regenerated cellulose the amount of the original sample dissolved ran as high as 12–13% which would appear to be too high to be caused by any other factor under the experimental conditions employed. Since cellulose xanthate exhibits this dispersive power, it at least makes it unnecessary that, when cellulose is xanthated, each cellulose chain contain xanthate groups before the material can be dispersed. Previous work has shown that solubility of cellulose xanthate appears a considerable time before complete penetration of the structure is possible. The chains on the surface of the microfibrils would therefore be substituted while those within would carry no xanthate groups. Such a structure has been shown to be soluble, and the solubility is ascribed to the dispersive action of the xanthated chains upon the unxanthated. This mechanism appears to be confirmed by the experimental results here reported.

Text. Research J. May 1946 Author

Chromatic Value Diagram

Modified chromatic value color space. J. L. Saunderson and B. I. Milner. *J. Opt. Soc. Am.* **36**, 36–42 (Jan. 1946).

In an effort to find an analytical transformation from I.C.I. notation to a uniform color space, the authors have developed a modification of the Adams' "chromatic value" diagram that serves further to smooth out and equalize on a single diagram the contour relationships between hue and chroma. The resulting "zeta space," like the "omega space" of Moon and Spencer, is based upon the recently published Munsell re-notations. Graphs and a special slide rule are described that make it a fairly simple matter to calculate color differences directly from I.C.I. data. The authors find their method equally as easy as using charts to obtain Munsell re-notations, and to have the advantage of eliminating errors due to inter-

polation. They expect to report later on extensive use of this color space for color-difference specifications. D. Nickerson

Text. Research J. May 1946

Crystallization of Polymers

The crystallization and crystallizing force of polymers. A. P. Aleksandrov. *Trudy Konferentsii Vysokomolekulyar. Soedineniyam, Akad. Nauk S.S.S.R., Otdel. Khim. Nauk i Otdel. Fiz-Mat. Nauk* **1**, 7–9 (1943) (pub. 1945) (through *Chem. Abstr.* **40**, 266[2] (Jan. 20, 1946)).

Many anomalous properties of polymers at temperatures below their melting point are attributed to formation of "fringes" around the crystals, composed of unoriented portions of polymer chains. These do not form part of the ordered crystal, and may be bound into another crystal in an unoriented manner.

Text. Research J. May 1946

Congo Red

Deposition of Congo red in cellulose. Oskar Wälchli. *Vierteljahrsschr. naturforsch. Ges. Zürich* **90**, 149–1 (1945) (through *Chem. Abstr.* **40**, 1032[9] (Feb. 20, 1946)).

In order to determine the position of deposits of Congo red in ramie fibers relative to the long axes of the fibers, the dyestuff in solid form and in solution, as well as the dyed fibers, was examined by various optical methods and x-ray analysis. Evaluation of the streaming birefringence data obtained with the Signer instrument from dye solutions in H_2O, 50% EtOH, and 50% glycerol makes it probable that the micellar aggregates in EtOH are composed of 7 to 8 and in glycerol of 2 to 4 single molecules and that the long axes of the latter, having dimensions of $24 \times 7 \times 3$ Å., lie parallel to the micelle axes and also parallel to the greatest *n*. X-ray analysis of the crystallized dye yielded a fiber diagram with an identity period along the long axis of the crystal of 24.03 Å. Comparison of this value with the length of the benzidine nucleus (21 Å.), together with an intermolecular distance of about 3 Å., as well as the evaluation of other data, justifies the assumption that the solid Congo red molecules are oriented with their long axes parallel to the long axis of the crystals. For all wave lengths, the dyed ramie fibers showed positive dichroism and an increase in birefringence, which was smallest for blue and greatest for green light. Cross sections of Congo red-dyed fibers showed that the glycerol solution containing the smaller micellar aggregates dyed at a much faster rate than the aqueous solution containing the larger aggregates. Obviously, the dye is colloidally adsorbed within the walls of the larger capillaries (diameter, 100 Å.) of the fibers with its single molecules oriented parallel to the fiber axis.

Text. Research J. May 1946

Enzymic Synthesis of Starch

The enzymic synthesis and degradation of starch. Part I. The synthesis of amylopectin. E. J. Bourne and S. Peat. *J. Chem. Soc.* **1945**, 877–82 (Dec. 1945).

The synthesis from glucose-l-phosphate of the unbranched amylose component of starch is catalyzed by a phosphorylase found by Hanes in potato juice. The authors have now isolated from the same source an enzyme (*Q*-enzyme) which, acting in conjunction with purified phosphorylase (*P*-enzyme), effects the conversion of glucose-l-phosphate into the major component of whole starch—namely, amylopectin. The synthetic amylopectin is nonreducing, does not retrograde from solution, stains red-purple with iodine, and is attacked by β-amylase in the manner characteristic of branched chain structures; i.e., hydrolysis proceeds with the liberation of maltose until a limit represented by a conversion of 46% is attained. Thereafter the action of β-amylase ceases. In these properties, the synthetic product is not to be distinguished from natural potato amylopectin. Furthermore, methylation of the synthetic amylopectin and end-group assay on the methyl ether show the unit chain to contain 20 glucose members. The probable course of the synthesis of whole starch in the plant is outlined and the respective roles of the *P*- and *Q*-enzymes therein are described. As an intermediate in the conversion of glucose-l-phosphate to amylose and amylopectin, the existence of a pseudo-amylose is postulated. Pseudo-amylose consists of an unbranched chain of 20 glucose units mutually linked by α-1,4-glucosidic bonds, as in amylose. Authors

Text. Research J. May 1946

Conversion of Amylose to Amylopectin

The enzymic synthesis and degradation of starch. Part II. The amylolytic function of the *Q*-enzyme of the potato. E. J. Bourne, A. Macey, and S. Peat. *J. Chem. Soc.* **1945**, 882–8 (Dec. 1945).

The *Q*-enzyme isolated from potato juice (*cf.* preceding abstract) effects a liquefying action upon starch paste and converts amylose (unbranched starch fraction) into amylopectin (branched starch fraction). Evidence is presented that its action is different from that of α- or β-amylases and that the best preparation is essentially uncontaminated with them. E. F. Evans

Text. Research J. May 1946

Fractionation of Nitrocellulose

The relation of nitrocelluloses of differing degrees of heterogeneity to volatile and nonvolatile solvents. S. N. Danilov. *Trudy Konferentsii Vysokomolekulyar. Soedineniyam, Akad. Nauk S.S.S.R., Otdel. Khim. Nauk i Otdel. Fiz.-Mat. Nauk* **1**, 36–7 (1943) (pub. 1945) (through *Chem. Abstr.* **40**, 459[2] (Jan. 20, 1946)).

Fractionation of nitrocellulose from Me_2CO and Et_2O, followed by precipitation with H_2O, separates apparently homogeneous esters into fractions with different N contents. The more soluble fractions have no peptizing action on the less soluble

ones. Fractions containing about 12% N show greatest and most rapid absorption of solvent vapors. Swelling pressure of the fractions after absorption of solvents depends on degree of nitration and temperature but not on viscosity of the solutions. Fractions which swell most have lower viscosities in solution and form more plastic resins.
Text. Research J. May 1946

Frictional Properties of Wool

The frictional properties of wool fibers. E. H. Mercer. *J. Sci. and Ind. Research* **18**, 188–200 (1945) (through *Chem. Abstr.* **40**, 467[4] (Jan. 20, 1946)).

A "stick-slip" method of studying the frictional properties of single wool fibers is described. The coefficients of friction varied with the pH of wetting solutions. The frictional difference between rootward and tipward motion increased in acid and alkaline media and this is associated with more rapid felting in these media. Several shrinkage-reduction treatments, the Cl, Br, sulfuryl chloride, and caustic alkali processes, were shown to reduce the frictional difference and this is the probable cause of the reduced rate of felting following these treatments.
Text. Research J. May 1946

Hydroxyethylcellulose

Sol-gel transformation of water-soluble ethylated hydroxyethylcellulose. Sven Sönnerskog. *Svensk Papperstidn.* **48**, 413–16 (Sept. 15, 1945) (in English) (through *Bull. Inst. Paper Chem.* **16**, 132 (Dec. 1945)).

Hydroxyethylcellulose may be ethylated more uniformly than cellulose to give water-soluble mixed ethers of greater commercial usefulness than that possessed by the low substituted (water-soluble) simple ethyl ethers. The mixed ethers are prepared by treating aged alkali cellulose from purified sulfite pulp with a mixture of ethylene oxide and ethyl chloride in an autoclave, first at 35° and later at 110°C. A series of samples of increasing combined ethylene oxide content was prepared and the temperature of hydration and the temperature of gelation (coagulation) of their aqueous solutions were determined. It was found that a straight-line relationship exists between these two characteristics for degrees of substitution from 0.15 to 0.75 moles of ethylene oxide per anhydroglucose unit. For mixed ethers of a degree of substitution of 1.5, prepared from alkali celluloses of increased ageing times, a simple relation was also found to exist between their viscosity in water and their gelation temperature, the latter increasing with decreasing viscosity (increasing ageing time for the alkali cellulose).
Text. Research J. May 1946

Mineral Khaki

Experiments on mineral khaki. I. S. R. Ramachandran and V. R. Wadekar. *J. Indian Chem. Soc., Ind. & News Ed.* **7**, 71–4 (1944) (through *Chem. Abstr.* **40**, 211[5] (Jan. 10, 1946)).

Six commercial samples of khaki fabrics, dyed by impregnation with Fe and Cr salts and their precipitation as hydroxides, contained 4.48–5.89% Cr_2O_3 and 0.59–1.24% Fe_2O_3. Light-fastness, rated 5–6 to 8 by the method of Bhat and Ramachandran (cf. *C.A.* **37**, 1043[5]), influenced favorably by the Fe content. The fastness to laundering (4–5%), Cl (3–5%), perspiration (4–5%), and to acid (1–3%) were determined by the methods of the German Commission on Fastness. The colors, on the Lovibond tintometer, were, in red-yellow-blue: 2.5–4.3–1.6; 1.8–3.3–1.3; 2.1–3.6–1.8; 2.9–3.8–2.3; 2.6–4.6–2.5; 2.7–4.7–2.4. Literature on khaki dyeing is reviewed.
Text. Research J. May 1946

Physical Properties of High Polymers

The relation of the physical properties of solutions of compounds of high molecular weight to temperature. V. A. Kargin. *Trudy Konferentsiĭ Vysokomolekulyar. Soedineniyam, Akad. Nauk S.S.S.R., Otdel. Khim. Nauk i Otdel. Fiz.-Mat. Nauk* **2**, 60–5 (1944) (pub. 1945) (through *Chem. Abstr.* **40**, 266[9] (Jan. 20, 1946)).

When polymers are cooled, they show loss of viscous flow (intermolecular shifts) and of elasticity (intramolecular shifts). If the polymer contains polar groups that react with one another, viscous flow may be lost before elasticity. These effects are more noticeable in solutions than in the pure polymers. Gel formation corresponds to loss of viscous flow. The temperature of gel formation in gelatin and other protein solutions can be raised by adding quinone to the solutions to form more intermolecular bonds. Gels show about the same deformation at all rates and there is little dependence on temperature, whereas the reverse is true for solution. Thus it is possible to determine the state of a polymer solution by measuring the physical properties. It is thus shown that nitrocellulose containing EtOH exists as a gel, whereas nitrocellulose and butyl phthalate form a solution. The same effect is noted with pure polyisobutylene that exists in a state corresponding to a solution. Addition of 5–6% Cl converts it to a gel-like state.
Text. Research J. May 1946

Polymers from Cellulose Derivatives

Copolymerization of unsaturated cellulose derivatives. S. N. Ushakov. *Trudy Konferentsiĭ Vysokomolekulyar. Soedineniyam, Akad. Nauk S.S.S.R., Otdel. Khim. Nauk i Otdel. Fiz.-Mat. Nauk* **1**, 35–6 (1943) (pub. 1945) (through *Chem. Abstr.* **40**, 458[5] (Jan. 20, 1946)).

Ethylcellulose is treated with ethylene oxide or ethylene chlorohydrin in the presence of alkali and the product is dehydrated with tetrachlorophthalic acid in xylene to give ethylvinylcellulose (*I*). Heating alkali cellulose with a mixture of ethyl chloride and allyl bromide in an autoclave gives mixed ethylallylcelluloses (*II*). *I* can be copolymerized with maleic esters and *II* with SO_2 to give products

whose physical properties vary with the number of unsaturated groups introduced into the cellulose molecule.

Text. Research J. May 1946

Polystyrene

Molecular-size distribution of cross-linked polystyrene. I. Valyi, A. G. Janssen, and H. Mark. *J. Phys. Chem.* **49**, 461–71 (Sept. 1945).

p,p'-divinyldiphenyl and *p,p'*-diisopropenyldiphenyl have been prepared. They are crystalline at room temperature but polymerize rapidly upon melting. Diisopropenyldiphenyl is stable at room temperature for a long time, while divinyldiphenyl polymerizes in the crystalline state within a few days. Copolymers of styrene with 10, 25, 100, and 1,000 p.p.m. of diisopropenyldiphenyl were made, and it was found that samples containing more than 300 p.p.m. are practically insoluble. Molecular-weight distribution curves for the soluble polymers were worked out. It was found that with increasing amount of cross-linking agent the weight-average DP shifts to larger values, the most frequent DP shifts to smaller values, and the distribution curve flattens out. Authors

Text. Research J. May 1946

Sodium Pectate Fibers

An x-ray diffraction investigation of sodium pectate. K. J. Palmer and Merle B. Hartzog. *J. Am. Chem. Soc.* **67**, 2122–7 (Dec. 1945).

Sodium pectate fibers were formed by forcing a partially neutralized solution of pectic acid through a nozzle into a coagulating bath of 85% ethyl alcohol 1*N* with respect to HCl. The resulting fiber was converted to sodium pectate by placing overnight in 0.1*N* 60% alcoholic NaOH. The excess salt was removed by 24 hours' immersion in 60% ethyl alcohol. The fiber was then removed and slowly stretched while drying in the air. Fibers prepared by this method gave excellent x-ray photographs. The fiber axis identity period is 13.1 Å. It has been shown recently that pectin is composed essentially of α-*d*-galacturonide units in the pyranose form connected by 1:4 glycosicidic linkages. Interpretation of the x-ray data presented is consistent with a galacturonide chain with a 3-fold screw axis and the closest packing arrangement. This fiber identity period is somewhat less than the value found for some cellulose derivatives in which the chain has the configuration of a 3-fold screw axis. This difference in identity period has been discussed in terms of molecular models. Sodium pectate at a relative humidity of 60% contains 18% water, mostly in the crystalline portion of the material. The non-uronide material (18%) is shown to have no detectable influence on the x-ray pattern. A. R. Macormac

Text. Research J. May 1946

Thermal Properties of High Polymers

The resistance to cold of high-molecular compounds. A. P. Aleksandrov. *Trudy Konferentsii Vysokomolekulyar. Soedineniyam, Akad. Nauk S.S.S.R., Otdel. Khim. Nauk i Otdel. Fiz.-Mat. Nauk* **2**, 49–59 (1944) (pub. 1945) (through *Chem. Abstr.* **40**, 266[7] (Jan. 20, 1946)).

The theory of reversible and irreversible deformation of macromolecules is discussed in terms of the internal forces involved. The effect of temperature on the physical properties of the polymers that do and do not form glasses is explained in terms of the theory.

Text. Research J. May 1946

Vulcanization of Cellulose

Vulcanization of cellulose materials. P. V. Afanas'ev and S. E. Bresler. *Trudy Konferentsii Vysokomolekulyar. Soedineniyam, Akad. Nauk S.S.S.R , Otdel. Khim. Nauk i Otdel. Fiz.-Mat. Nauk* **2**, 120–9 (1944) (pub. 1945) (through *Chem. Abstr.* **40**, 458[9] (Jan. 20, 1946)).

Alkylation of cellulose by compounds of the type $ROCH_2{-}NR_3Cl$ (*I*) is due to formation of positively charged ions of $—O{:}CH_2$ which are stabilized by the occurrence of resonance forms. If aromatic rings or chains of conjugated double bonds are introduced, the ions are more stable and can react with OH groups in cellulose under milder conditions than can *I* itself. Such compounds are obtained by condensing pyridine-HCl with the bis-(chloromethyl) ether of diethylene glycol, bis(2-chloroethyl) ether, the tris(chloromethyl) ether of glycerol and the bis(chloromethyl) ether of decamethylene glycol. Viscose fibers are soaked in aqueous solutions of these compounds for 10 hrs. and then heated at 120° for 20 min. Vulcanization occurs and the products swell less in solvents than the original fiber. Partly hydrolyzed polyvinyl acetate and polyvinyl alcohol undergo the same reaction.

Text. Research J. May 1946

Xanthic Acid Derivatives

The chemistry of xanthic acid derivatives. Part I. The preparation and comparative properties of isomeric xanthates and dithiocarbonates. G. Bulmer and F. G. Mann. *J. Chem. Soc.* **1945**, 666–74 (Oct. 1945).

The preparation and properties of several series of isomeric compounds consisting of two xanthates and one dithiocarbonate, of structure RO··CS·SR′, R′O·CS·SR and RS·CO··SR′ respectively, have been studied. The action of phenylhydrazine differentiates decisively between the three isomerides, for the two xanthates furnish different thiourethanes, and the dithiocarbonate is unaffected. The pyrolysis of these compounds and their reaction with chloramine-T have been studied and are discussed. E. F. Evans

Text. Research J. May 1946

The chemistry of xanthic acid derivatives. Part II. The thermal decomposition of disulfurdicarbothionic esters. G. Bulmer and F. G. Mann. *J. Chem. Soc.* **1945**, 674–7 (Oct. 1945).

The dialkyl and various dibenzyl disulfurdicarbothionates, $(ROCSS)_2$,

usually can be readily prepared by the action of chloramine-T or sodium tetrathionate on the corresponding alkali xanthate. The diethyl and dimethyl esters distill unchanged when heated at 0.1 mm. pressure, but at about 17 mm. pressure undergo thermal decomposition to the corresponding xanthate, ROCSSR. The dibenzyl ester, and di-*p*-methyl and di-*p*-chloro analogues, however, when heated at 0.1 mm. undergo decomposition to the corresponding disulfides, while at 18 mm. or higher pressures they furnish the corresponding stilbenes; the corresponding xanthates are apparently never formed. It is shown that the formation of the stilbenes in these reactions is almost certainly independent of that of the disulfides, although the latter may also furnish the stilbenes under suitable conditions. Authors

Text. Research J. May 1946

The chemistry of xanthic acid derivatives. Part III. The interaction of carboxylic acid chlorides and potassium ethyl xanthate. G. Bulmer and F. G. Mann. *J. Chem. Soc.* **1945**, 677–80 (Oct. 1945).

The few *O*-ethyl-*S*-(carboxy)acylxanthates previously recorded have been isolated only as unstable liquids. It is now shown that *p*-nitrobenzoyl chloride reacts with potassium ethyl xanthate to form the crystalline *O*-ethyl-*S*-*p*-nitrobenzoyl xanthate, which can be recrystallized unchanged: the *p*-chlorobenzoyl analogue is similar. The thermal decomposition products of each have been investigated. *O*-ethyl-*S*-benzoyl xanthate is, however, an unstable liquid which undergoes violent thermal decomposition affording initially dibenzoyl disulfide and ultimately a mixture of ethyl benzoate, thiobenzoate and dithiobenzoate, and 2,3,4,5-tetraphenyl-thiophen nonasulfide, the properties of which have been studied. Xanthates thus obtained from carboxylic acid chlorides are, however, more stable than those from sulfonyl chlorides, which apparently undergo spontaneous decomposition on formation.

Text. Research J. May 1946 Authors

The chemistry of xanthic acid derivatives. Part IV. The interaction of sulphonyl chlorides and potassium ethyl xanthate. G. Bulmer and F. G. Mann. *J. Chem. Soc.* **1945**, 680–6 (Oct. 1945).

Potassium ethyl xanthate reacts with benzene-, *p*-toluene-, and naphthalene-2-sulfonyl chlorides to form the corresponding potassium sulfonate, the disulfone RSO_2SO_2R, and diethyl disulfurdicarbothionate, $(EtOCSS)_2$. In the reaction with other sulfonyl chlorides, the production of the disulfone may be partly or wholly replaced by that of the thiolicsulfonate RSO_2SR, the disulfide RSSR, or the corresponding sulfinic acid. Evidence is adduced that the initial reaction in all cases is the formation of the unstable, highly reactive sulfonyl xanthate, $EtOCSSSO_2R$; the mechanism by which the above products arise from this xanthate is discussed. Many alkali xanthates (and hence carbon disulfide itself) can be readily identified by conversion to the corresponding palladium xanthates, $(ROCSS)_2Pd$, which are non-ionic compounds usually having characteristic melting points. Authors

Text. Research J. May 1946

BLEACHING: DYEING: FINISHING

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Dyeing Narrow Fabrics

The package-dyeing of narrow fabrics: A link between yarn dyeing and piece dyeing. H. A. Thomas. *J. Soc. Dyers and Colourists* **61**, 185–91 (Aug. 1945).

The various methods available for dyeing narrow fabrics (up to 18 in. in width) are described, and their merits in comparison with the manufacture of webs from dyed yarns are assessed. Many advantages, both technical and on a cost basis, result from the preparing, dyeing, and finishing of such fabrics in yarn package-dyeing machines; this has been demonstrated on a large scale. All cotton webbing dyed in roll form with direct dyes and with sulfur black is thoroughly penetrated; other sulfur colors and the azoic dyes have not yet been used successfully; heavy webbing dyed with vat dyes is not completely penetrated, but the shade is level and the penetration is adequate for the purpose. Discovery of the principle of slack rolling of the narrow fabric which permits channeling of dye liquor between laps (and is contrary to accepted doctrine in pressure-dyeing) was necessary for the success of this development. Details of many plant dyeing procedures are presented. K. S. Campbell

Text. Research J. May 1946

History of Direct Dyes

The diamond jubilee of the discovery of direct cotton dyes—1884–1944. C. M. Whittaker. *J. Soc. Dyers and Colourists* **61**, 201–3 (Aug. 1945).

A chronological survey discussing briefly the properties of a number of direct cotton dyes the introduction of which marked definite progressive steps in the expansion of this group. Some of the earliest direct dyes—Benzopurpurine 4B (1885), Black BH (1890), and Sky Blue FF (1891), for example—are still widely used today. K. S. Campbell

Text. Research J. May 1946

Oxidation of Cellulose During Vat Dyeing

The atmospheric oxidation of cellulose in presence of reduced vat dyes. J. Brear and H. A. Turner. *J. Soc. Dyers and Colourists* **61**, 273–8 (Nov. 1945).

In order to determine conditions affecting atmospheric oxidative modification of cellulose in the presence of reduced vat dyes, experiments were conducted in which the reduction-oxidation cycle (hydrosulfite-air) was repeated a number of times, and during which the pH at which reoxidation occurred was controlled at several levels; 21 vat dyes covering a wide range of known activity were used. To eliminate accelerating action by light, the work was done entirely in the dark. Damage was assessed by cuprammonium fluidity measurements. It

was revealed that for almost every dye examined each reduction-oxidation cycle caused an increase in fluidity of the cotton. In general the greatest rise in fluidity was brought about by those dyes which are active in promoting tendering by light. The maximum effect was observed in the region of pH 11.6 in all cases; Cibanone Orange R, which was examined in more detail, was shown to be more active in tendering when the temperature of reoxidation was raised, also when the reoxidation was carried out in sunlight. A method of preparing cuprammonium solution for fluidity determination using cuprous oxide is appended. K. S. Campbell

Text. Research J. May 1946

Mineral Khaki

The dyeing of cotton with mineral khaki. Part V—An examination of the factors which govern variations in the chromium and iron contents of yarns dyed by the mineral khaki process. E. Race, F. M. Rowe, and J. B. Speakman. *J. Soc. Dyers and Colourists* **61**, 224–33 (Sept. 1945).

It is shown that, as far as production of chromium and iron pigments on the fiber is concerned, nothing is gained by the use of mixtures of sodium hydroxide and sodium carbonate in the mineral khaki developing bath. Variations in the total oxide content of mineral khaki-dyed yarn are dependent upon variations in the amount of chrome-iron liquor retained by the yarn after impregnation and also upon the concentration of the alkali developing liquor. Careful chemical control of this liquor (simplified by the use of sodium carbonate alone) may rectify the oxide content variations caused by unequal pressures applied during hand-wringing. The carbonate alone as developing agent rather than sodium hydroxide is also preferred because the finished yarn is much less likely to retain alkalinity to phenolphthalein. In hydroxide-carbonate developing baths containing progressively increasing proportions of carbonate (as occurs in ordinary practice), iron oxide tends more and more to be preferentially precipitated on the yarn, while the chromium salt in solution on the yarn, being precipitated more slowly, tends to pass into the developing liquor and to be precipitated there rather than on the fiber. It is stated, by way of introduction, that it is now known that alkali-soluble chromium has not the significance with respect to combating attack by micro-organisms which it was thought to have when the work reported was carried out. K. S. Campbell

Text. Research J. May 1946

The dyeing of cotton with mineral khaki. Part VI—A method of limiting the variations in oxide content of mineral khaki-dyed yarn by chemical control of the developing bath. E. Race, F. M. Rowe, and J. B. Speakman. *J. Soc. Dyers and Colourists* **61**, 233–6 (Sept. 1945).

The use of a concentration of sodium carbonate in the developing bath not greatly in excess of the stoichiometric amount required for complete precipitation of chromium and iron held by the impregnated yarn, together with the maintenance of this concentration by controlled additions of sodium carbonate, leads to the production of a mineral khaki-dyed material which does not require subsequent washing or treatment in mildly acid solution provided that the final wax treatment is carried out in a slightly acid medium. When the concentration of developing liquor is kept constant, the chromium, iron, and alkali-soluble chromium contents of the yarn are also constant.

Text. Research J. May 1946 Authors

Processing Quality of Dyed Wool

The effect of dyestuffs on combing and spinning results. Anon. *Text. Recorder* **62**, 44–5 (Oct. 1945).

The experiments described were carried out to obtain evidence which would or would not support the view that slubbing-dyed wool is not as satisfactorily processed as undyed wool, and that dark shades give a poorer yield than light shades. Curves and tables show the results. The dyed yarns had lower strengths and elongation; however, they were more uniform. A. L. Merrifield

Text. Research J. May 1946

Fabric Preservation with Naphthenates

Naphthenates in wood and fabric preservation. M. D. Curwen. *Oil Colour Trades J.* **109**, 82, 84, 86 (1946) (through *Chem. Abstr.* **40**, 1662[1] (Mar. 20, 1946)).

Cu naphthenate made rapid strides in replacing other wood and fabric preservatives. The slow leaching-out on exposed parts of materials treated with Cu naphthenate can be overcome by the part-formation of Al naphthenate during the process of manufacture. This mixture permits the estimated use of about 8% less naphthenate than is normally recommended.

Text. Research J. May 1946

Machines for Dry-Processing

Machines for dry-processing. Anon. *Text. Recorder* **62**, 46–9 (Oct. 1945).

The concluding article on the survey of machines employed for the dry-processing of fabrics. Softening, brushing, raising, shearing, napping, and pressing machines are described. 21 diagrams and figures illustrate the different machines reviewed. A. L. Merrifield

Text. Research J. May 1946

Resin-Finishing

The future of finishing. R. J. Smith. *J. Soc. Dyers and Colourists* **61**, 269–73 (Nov. 1945).

Particular emphasis is laid on the trend toward durability in textile finishes. Synthetic resins useful for textiles are divided schematically into thermosetting, including aldehyde condensates and alkyd resins, and thermoplastic, divided into the vinyl types, the acrylate types, polythene, and polyamides. A concise description of the properties of each important resin is presented and their finishing possibilities are discussed. Cellulose ether finishes,

outstanding for durability and certain to become important, are described. K. S. Campbell

Text. Research J. May 1946

Slashing Problems

Trouble spots in warp slashing. H. B. Sweat. *Rayon Text. Mo.* **27**, 601–2 (Nov. 1945).

A number of troubles commonly encountered in slashing are described and remedies suggested. E. G. Martin

Text. Research J. May 1946

Urea Derivatives

Urea derivatives instead of urea for textile treatments. Anon. *Silk and Rayon* **19**, 1220–1, 1223–4 (Nov. 1945).

The second of a series. A brief outline and discussion of the manufacture and use of the following are given: (1) phenylguanazole for flameproofing textile materials (B.P. 564,573), (2) bonding of plywood with phenylguanazole-formaldehyde resins (B.P. 565,490), (3) increase of the affinity of cellulose materials for acid wool dyes (B.P. 429,209 and 565,675), (4) mono-guanidinium, bis (guanidinium) and tris (guanidinium) compounds for crease-resistants, increased fastness of direct dyes on cellulose, and dyeing cellulose with acid and chrome dyes, (5) increased fastness of the guanidine-formaldehyde resin on the cellulose by addition of resorcinol (B.P. 560,121). A. L. Merrifield

Text. Research J. May 1946

FIBERS: YARNS: FABRICS: MECHANICAL PROCESSES

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Acetylated Cotton

Acetylated cotton highly resistant to rotting. Charles F. Goldthwait and Samuel T. Voorhies, Jr. *Textile World*, **96**, 115–17, 212, 216 (Feb. 1946).

Partial acetylation can be achieved on fiber, yarn, thread, or cloth to forestall microbiological attack. The character of the cloth is said to be unaffected. Uniform treatment is admitted to be difficult to attain in heavy, tightly woven fabrics and treatment of yarns is recommended for these fabrics. Extensive tests, as yet incomplete, indicate unusual resistance to rotting and mildewing under conditions so severe as to rot completely ordinary cotton in about a week. H. J. Burnham

Text. Research J. May 1946

Structure of Animal Fibers

Animal fibers. Part 1. Their growth and structure. J. L. Stoves. *Fibres* **7**, 5–7 (Jan. 1946).

A review of present knowledge of the growth, chemical constitution, and microstructure of animal fibers. Photo-micrographs are given of kemp hair from Icelandic sheep, hare fibers, calf hair, and fur seal. 20 references. A. R. Martin

Text. Research J. May 1946

Cotton Fiber and Yarn Relationships

Relationships between properties of cotton fibers and appearance of carded yarns. Robert W. Webb and Howard B. Richardson. Cotton Branch, Production and Marketing Administration, U.S.D.A. Pp. 1–47, Figs. 1–14 (Mar. 1946). (Processed.)

This report completes the fourth segment of a broad study on the relationships of cotton fiber properties to manufacturing performance and quality of manufactured products. The findings presented refer principally to the appearance of 22s and 60s carded yarn manufactured from case-historied American Upland cottons. Supplemental studies having more particular reference to fiber maturity have been conducted on the appearance of 22s and 60s carded yarn for these cottons and for those of the 1944 annual variety series; and on the appearance of 60s and 100s combed yarns for a series of long-staple cottons, including Sea Island, American-Egyptian, and Upland growths, 1941–44. A total of approximately 1,100 selected cottons and 1,500 lots of yarn are involved in the studies reported. Multiple, partial, and simple correlation analyses have been made, as in the earlier studies, the emphasis being given to the comparative significance of alternative fiber length and strength measures, in terms of appearance of 22s carded yarn. The fiber properties included are upper-quartile length and coefficient of length variability (sorter), upper-half mean length and length-uniformity ratio (fibrograph), strength (Chandler and Pressley), fineness, percentage of mature fibers, and grade. Mean fiber length, as determined by both laboratory methods, and classer's staple length have been considered in some particulars. For the relationship existing between the appearance of 22s carded yarn and the 6 collective fiber properties, the coefficient of multiple linear correlation is 0.820 when sorter length and Chandler strength measures are used, and 0.784 when fibrograph length and Pressley strength measures are used. The coefficients of determination indicate that 67.3% of the variance in the appearance of 22s carded yarn made from these cottons is accounted for by the 6 fiber properties, when sorter length and Chandler strength measures are used, as compared with 61.5% by the 6 fiber properties, where fibrograph length and Pressley strength measures are used. Multiple curvilinear correlation analyses have failed to give any higher correlation. Almost as much variance in yarn appearance is accounted for by the 3 collective fiber properties of grade, upper-quartile length (sorter), and coefficient of length variability (sorter) as is obtained with 6 fiber properties. The 2 fiber properties of grade and length-uniformity ratio (fibrograph) give practically as high a correlation with yarn appearance as 6 fiber properties in combination. Grade and coefficient of length variability (sorter) account for 57.9% of the variance in yarn appearance. Upper-quartile length and coefficient of length variability explain 57.6% of the yarn-appearance variance. Grade and staple length account for 50.1% of the variance in the appearance of these yarns. Upper-half mean length and uniformity ratio in combination, as determined by

the fibrograph method, account for 47.9% of the variance in the appearance of these yarns. The results of simple correlation analyses indicate relatively low or poor correlation between any one of the fiber properties and yarn appearance. Coefficient of length-variability (sorter), length-uniformity ratio (fibrograph), and grade give the best results. On the basis of the findings from 2 sets of partial correlation analyses identified with the alternative fiber length and strength measures, the significantly contributing fiber properties rank in order of net importance to yarn appearance, as follows: coefficient of length-variability (sorter), grade of cotton (classer), upper-quartile length (sorter); and grade of cotton (classer), length-uniformity ratio (fibrograph), fiber tensile strength (Pressley). The other measured fiber properties, namely, upper-half mean length (fibrograph), strength (Chandler), fineness, and percentage of mature fibers, show no significant contribution to yarn appearance. Over 40 regression equations are presented in this report as the basis for revealing the relationships existing between the appearance of 22s carded yarn and variously combined and separate fiber properties. These equations also serve as the basis for estimating yarn appearance from a knowledge of the magnitudes of the fiber properties considered. Relatively precise estimates are possible by the several best equations, even though comparatively low correlation exists. In the light of these results, it is possible that some fiber property or properties not included in this analysis may be matters of importance to yarn appearance. Or, it may be that factors and variables in the manufacturing organization and drafting processes, even when held constant or comparable for different lots of cotton, may contribute significantly to yarn appearance by differential action on different cottons. Limitations and variations in the precision of present-day methods for evaluating different degrees of yarn appearance, as well as for measuring the fiber properties, also may affect the end-results to some extent. In considering the statistical equations presented in this report for estimating yarn appearance, it should be remembered that they refer to American Upland cottons and to the appearance of 22s carded yarns. The relationships between fiber properties of long-staple cottons and yarn appearance in various counts of combed yarn differ in some particulars from those here reported. Authors

Text. Research J. May 1946

Defibering Pita

Chemical method for the industrial defibering of the *Bromeliaceae*. Enrique Pérez Arbeláez. *Rev. acad. columbiana cienc. exactas fis. nat.* **6**, 235–43 (1945) (through *Chem. Abstr.* **40**, 1319³ (Mar. 10, 1946)).

Methods are described for treating various South American species of this family of plants in preparing cordage and textiles for their fibers. Costs of operation and other aspects of commercial development in Columbia are considered. Mechanical and biological methods of separating the fibers have many disadvantages. The chemical method of the author consists mainly of heating the plant to 100° in water for 1 hr. to break up the cement between the fibers. The method has mainly been applied to *Bromelia magdalenae* Wright ("pita"). Other species have been tested experimentally. 20 references.

Text. Research J. May 1946

Flax

Flax can be run on cotton and worsted machinery. S. A. G. Caldwell. *Textile World* **96**, 106–7, 200 (Jan. 1946).

Manufacturing costs are much less when stapled flax fiber is run on cotton or worsted machinery rather than on conventional flax machinery. Cottonization, the key to utilizing flax fiber, is effected on a multiple-roller machine of the horizontal type used for jute softening. The degumming process, involving a wet treatment with chemicals to remove residual gums and other matter, is said to be most important and may involve a series of treatments with solutions of a sodium carbonate or hydroxide, alternating with or followed by immersion in a bath containing sulfuric acid. Moisture content should be kept to about 10–12%. Fibers are opened on a hard-waste opener followed by a single-process opener. Laps of flax and cotton may be blended on the finisher picker. The stock is then put through cards, draw frames, roving, and spinning in a conventional manner. In running flax on worsted machinery, fiber lengths of 4–6 in. are used, and standard worsted machinery is said to be suitable. H. J. Burnham

Text. Research J. May 1946

Organic Acids in Raw Cotton

The organic acid content of raw cotton fiber. Isolation of *l*-malic acid and citric acid from cotton fiber. Elizabeth R. McCall and John D. Guthrie. *J. Am. Chem. Soc.* **67**, 2220–1 (Dec. 1945).

Raw cotton fiber contains about 0.5% *l*-malic acid and 0.07% citric acid. The presence of both of these acids in raw cotton fiber has been definitely established by isolating them in crystalline form.

Text. Research J. May 1946 Authors

Woolen Top

Producing an even top. W. H. Robinson. *Text. Recorder* **62**, 43–4 (Nov. 1945).

A review of the factors to be considered in the production of woolen top to insure regularity of the finished sliver. The carding, backwashing, strong boxes, combing finishing, finishing gills, and drafting of the wool fiber for best results are described. A. L. Merrifield

Text. Research J. May 1946

Zein Fibers

Zein fibers—preparation by wet spinning. C. B. Croston, C. D. Evans, and A. K. Smith. *Ind. Eng. Chem.* **37**, 1194–8 (Dec. 1945).

A new method for producing textile fibers from zein and the equipment used in the process are described. Zein fibers which have high tensile

strength and wool-like properties are produced from alkaline dispersions of zein. The spinning dispersions are formulated to give a product of high viscosity by the use of denaturing agents, such as alcohol or urea, or by ageing the solutions. The filaments are coagulated in an acid bath in which salts may or may not be used. The coagulated filaments are given a mild formaldehyde precure prior to stretching and drying. The influence of the extent of the precure on the load-elongation properties of the fiber and on fiber strength is determined. Shrinkage and water-resistance of the fibers are controlled by acetylation followed by a strong formaldehyde treatment. Fibers with a dry strength of 1.87 and a wet strength of 0.75 g. per den. have been obtained. The dry strength of a good grade of wool is about 1.3 grams per den. Dry strengths on other synthetic protein fibers have been 0.6 to 0.8 g. per den. Fibers finer than 0.6 den. have been prepared. The zein fibers have high resilience and are very flexible. Acetylated and properly cured fibers showed excellent resistance to boiling in buffered solutions of pH 8–9.

A. R. Macormac

Text. Research J. May 1946

Woolen Yarn Twist

Woolen yarn twist. Henry L. Pero. *Am. Wool and Cotton Reptr.* **60**, 12, 39 (Feb. 28, 1946).

A report on the study of the relationship of twist to the strength and length shrinkage of woolen yarn made at Lowell Textile Institute on $2\frac{3}{4}$-run yarn. With values of turns per inch varying from 4.16 to 19.74, the strength reaches a maximum at about 11 t.p.i. while the critical point on the twist-shrinkage curve is reached at approximately 16 t.p.i.

H. J. Burnham

Text. Research J. May 1946

Damage to Fabrics during Fumigation

Damage to fabric by liquid hydrocyanic acid gas in fumigation. G. C. Sherrard. *U. S. Pub. Health Repts.* **60**, 1308–9 (1945) (through *Chem. Abstr.* **40**, 741[7] (Feb. 10, 1946)).

Text. Research J. May 1946

Experimental Milling Machine

The determination of milling and felting shrinkage on small samples of fabrics and loose fiber. N. H. Chamberlain and J. Menkart. *J. Soc. Dyers and Colourists* **61**, 286–91 (Nov. 1945).

Methods available for the determination of the milling shrinkage of small samples of fabric and loose wool are reviewed, and illustrative figures obtained by the hand-milling method are quoted. A new experimental milling machine is described, and typical test results obtained both with wool fabrics and loose wool samples are given. The machine is applicable to both soap and acid milling, and the limiting factor in the accuracy obtainable is probably the inherent sample-to-sample variation of the original material, rather than irregularities in the behavior of the mechanism.

Text. Research J. May 1946 Authors

Tender Warps

The weaving of tender and oversized woolen warps. Anon. *Text. Recorder* **62**, 54–5 (Dec. 1945).

The causes of tender woolen warps and ways to overcome the difficulties encountered during weaving of tender and oversized warps are discussed. A. L. Merrifield

Text. Research J. May 1946

Washing Woolen Fabrics

Suggestions on washing woolen fabrics. Anon. *Am. Wool and Cotton Reptr.* **60**, 13, 14 (Jan. 24, 1946).

A description of washing and fulling operations on woolen fabrics with suggestions on soaps and washing times. H. J. Burnham

Text. Research J. May 1946

Rayon Crepes

Problems in weaving rayon crepes. J. W. Hutchinson. *Silk and Rayon* **19**, 1106, 1109, 1111 (Oct. 1945).

The weaving of rayon crepes is an exacting task which presents many problems and depends to a large extent on the proper care and setting of the loom for weaving this type of fabric. The problems discussed are: the number of turns twist per in. in the yarn, twist setting, shuttle race, crank arms, breastbeam, back rail, tappet, shed and lease rods, heald shaft, drop box, circular box, shuttle, and temple. Figures illustrate the tappets, temple, and settings of open shed with crank at back center, and position of the shafts with the crank in 2 other positions. A. L. Merrifield

Text. Research J. May 1946

Carding Developments

Carding developments in relation to modern spinning technique. Anon. *Text. Recorder* **62**, 40–1 (Dec. 1945).

The present tendency is to reduce the number of doublings; and, therefore, where short-cut processes are contemplated more consideration must be given to carding in order to minimize the inherent defects in the machine and the sliver produced. The sources of sliver irregularity can be minimized by continuous stripping and by the use of non-flexible cylinder wire. In both cases stripping need be done only after relatively long periods of carding, thus reducing sliver irregularities, waste, and labor. A table and curves show the rate of cotton absorption by the cylinder, and the rate of sliver output with time. During the first 20 to 25 min., the sliver varies erratically, and a constant rate of variation is not attained until after 2 hrs. of operation. A. L. Merrifield

Text. Research J. May 1946

Cotton Card Attachment

The sliver reducer—something new for the carding department. Anon. *Textile Bulletin* **69**, 33–4 (Feb. 15, 1946).

A 3-roll drafting element to replace the conventional calendar roll assembly on a cotton card is described.

The total draft is about 2. It is claimed that one drawing process can be eliminated and that card production is increased from 10 to 30%. It is also said that greater fiber parallelization results in more sliver in the cans. No change in the final quality of yarn or fabric is noted. H. J. Burnham

Text. Research J. May 1946

Cotton Spinning Process

The cotton spinning process. Eugene C. Gwaltney. *Text. Recorder* **63**, 40–1 (Jan. 1946) and 41–2 (Feb. 1946).

There has been during recent years a great interest shown in the manufacture of yarns from fibers such as rayon, wool, etc., by the cotton process because of the tremendous saving offered by this system. The adaptability of the cotton spinning process to spinning of synthetics and wool fibers is discussed. Except for the presence of static electricity, which is more common in synthetic fibers than in cotton, the spinning of synthetic fibers presents fewer problems than cotton. If and when it is found that spinning wool on the cotton system is practical and profitable, then changes in dimensions of the machines will be made to accommodate staple lengths greater than 4 in., as no new principles are involved. A. L. Merrifield

Text. Research J. May 1946

New Automatic Loom

New automatic bobbin-changing loom for weaving rayon crepes. Anon. *Text. Recorder* **63**, 36–7, 43 (Jan. 1946).

A new 2 × 2 rayon crepe loom is described. This loom has many innovations, some of which are: the magazine is of the fixed type, electro-mechanical feeler blade, wooden shuttle box backs, take-up motion incorporates an 11-inch-diameter, rubber-covered roller, more sensitive let-off motion, speed control of warp beam, warp stop motion of 6-bar type, shuttle eye thread cutter, shuttle contains self-threading spring-tension eye, and a self-contained electric drive. The recommended speed for 2 × 2 crepes on a 48-in. loom is 150 picks per min., and single shuttle loom of similar type, 165 picks per min. A. L. Merrifield

Text. Research J. May 1946

Shuttle Selection and Setting

Careful selection and setting of shuttles is important. J. W. Hutchinson. *Silk and Rayon* **19**, 1358–9 (Dec. 1945).

The selection of shuttles and the setting of the shed, box front, etc., for best weaving results are discussed. Diagrams show the correct position of the shuttle to reed and box front, "off" delivery, "up" delivery of the shuttle, and views of wear at back and front of shuttle. The straightest-grained shuttles with the least number of knots should be employed for weaving the most costly warps and the most complicated designs. A. L. Merrifield

Text. Research J. May 1946

WAR RESEARCH ON TEXTILES

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U. S. Research

Coated Fiberglas

Flexibility test of coated fiberglas material. (Army Air Forces. Engineering Division. TSEAL-5F-672-9). F. I. Burstein. O.P.B. Report PB 5747, 1944; 1 p.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 459 (Mar. 15, 1946)).

Samples of fiberglas materials numbers 116, 162, and 164, neoprene-coated, and samples of fiberglas materials numbers 116, 128, and 162, vinyl-resin-coated, were tested and found suitable for use in equipment to be used in Arctic regions.

Text. Research J. May 1946

Flame-Resistant Suits

Close-out Project T-17, fabric, fireproof. (Army Air Forces. Personal Equipment Laboratory. TSEAL-5H5-4-198). John S. McGilly. O.P.B. Report PB 5106, 1945; 8 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 459 (Mar. 15, 1946)).

Tests were conducted on summer flying suits treated for flame retardency. Additional tests were conducted on various fiberglas fabrics and compounds of fiberglas and other fibers as asbestos and fireproof cotton. Results indicate that the untreated summer flying suit offers protection against flash fires of short duration. Fires of over 5 secs. will produce burns on the body beneath either treated or untreated suits. Heat-transfer through a fireproof fabric is sufficient for a flame of short duration to produce severe burns beneath fireproof fabric. Photographs.

Text. Research J. May 1946

Milkweed-Filled Flying Suit

Milkweed-filled flying suit. (Army Air Forces. Engineering Division. ENG-54-666-83B.) F. A. Maham. O.P.B. Report PB 5732, 1943; 2 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 345 (Mar. 8, 1946)).

The object of the work described in this report was to furnish information concerning the inspection and tests of a milkweed-quilted flying suit. It was concluded that the quilted suit of milkweed is not satisfactory as a garment to be worn by personnel subjected to low temperatures and is inferior to Army Air Forces cold-weather clothing. The construction of the garment made it impossible to test the merit of the milkweed floss. Previous tests on milkweed for Army Air Forces clothing indicated that the milkweed filler was highly inflammable.

Text. Research J. May 1946

Air Permeability Tests

A method for studying the effect of wind on the insulation value of windbreak and other fabrics. (Quartermaster Corps Climatic Research Laboratory Report 43.) Richard L. Day. O.P.B. Report PB 3415, 1943; 13 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 334 (Mar. 1, 1946)).

Description of a method for determining the actual loss of insulation caused by the penetration of wind into a given fabric combination in a physical test in which the conditions resemble those present when the fabric is being worn. Fabric is wrapped around an electrically heated copper cylinder, and air speeds up to 1,350 feet per min. are provided by a wind tunnel. It is concluded that a good windbreak, such as poplin or sateen, provides adequate protection against wind. Contains statistical diagrams.

Text. Research J. May 1946

Underwear Tests

Test of new special underwear (white) to determine shrinkage and stretch on impregnation and wearing. (Chemical Warfare Service. Technical Command Informal Report T.C.I.R. 69). B. Berger. O.P.B. Report PB 5034, 1944; 19 pp.; microfilm, 50¢—photostat, $2.00 (through *Bib. Sci. and Ind. Reports* **1**, 407 (Mar. 15, 1946)).

A description of 3 tests made to evaluate changes in special cotton underwear procured in recent years under Q.M. Corps Tentative Spec. P.Q.D. Nos. 39 and 40. The general procedure of the test consisted of subjecting the underwear to 2 cycles of impregnation, wear, and laundering and determining the shrinkage and fitting qualities of the garments during processing. Comparison of the changes in measurements are given in tables in Appendix *A*. Photographs show the fitting qualities of the 3 styles. Results prove that the underwear manufactured in accordance with the Quartermaster Corps Tentative Specifications P.Q.D. No. 39C, and P.Q.D. No. 40C will have adequate fitting qualities after impregnation, servicing, and wear, provided the undershirts have double-layer sleeve cuffs.

Text. Research J. May 1946

Scandinavian Research

Scandinavian textile research and education. Julian F. Smith. O.P.B. Report PB 3888, 1945; 13 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and and Ind. Reports* **1**, 387 (Mar. 8, 1946)).

This report on textile laboratories visited in Sweden and Denmark contains sections on the following subjects: (1) fundamental research on high polymers; (2) industrial research on cellulose and textiles; (3) paper yarn as a textile product; (4) textile education in Sweden; and (5) testing and standardization. Institutions visited include: Physicochemical Institute at the University of Uppsala; Dr. Erik Hägglund's laboratory at the Kungl. Teknisk Högskola (Royal University) of Stockholm; Danish Textile Research Institute; and Danish Technical Library, Copenhagen.

Text. Research J. May 1946

German Research

Circular Knitting

Circular knitting and its development in Germany since 1930. P. H. Hanes, Jr., and C. L. Rosenquist. O.P.B. Report PB 6347, 1945; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 459 (Mar. 15, 1946)).

This report contains sections on the following subjects: knitting yarns, development of circular interlock knitting, description and technical details of the Fouquet & Franz circular interlock machine, and circular knitting machine development since 1935. Appendix *A* lists 34 names of companies visited, location, product manufactured, and number of employees before the war.

Text. Research J. May 1946

Cotton Exports to Germany

Factors relating to prospects for exporting U. S. cotton to Germany (with limited information on Italy and Switzerland). Robert C. Jackson. O.P.B. Report PB 4495, 1945; 44 pp.; microfilm, 50¢—photostat, $3.00 (through *Bib. Sci. and Ind. Reports* **1**, 333 (Mar. 1, 1946)).

An analysis of the German textile industry during the war, its present status and future prospects, with particular reference to possible markets for American cotton. Future consumption of U. S. cotton depends on such factors as exchange (ability of Germans to have acceptable exchange or credits with which to make payments), competition with the extensive synthetic fiber industry developed under the Third Reich, competition with other nations producing cotton, and quality and packaging of raw cotton. Investigator believes all these problems can be solved. A brief report is also made on the possible cotton markets open in Italy and Switzerland. Report contains list of cotton merchants and cotton agents in Bremen area.

Text. Research J. May 1946

Documents

Documents relating to textile research, *Melliand Textilberichte*, Heidelberg. Julian F. Smith. O.P.B. Report PB 3876, 1945; 2 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 387 (Mar. 8, 1946)).

This report lists: (1) outstanding researches to be found in *Melliand Textilberichte;* (2) testing and research developments; and (3) documents. Subjects of the documents listed are: new yarn-uniformity test method, bursting-strength tests of fabrics, influence of organic synthesis on the textile industry, spinning staple fiber on cotton machinery, blending wool with staple fiber in the woolen and worsted industry and finishing the fabrics, staple fiber and its blends with wool in carpet yarns, and equalizing thread tension in spinning machines by controlling rate of rotation.

Text. Research J. May 1946

Hollow Fiber Production

Hollow fiber, cellulose production, and stoll abrasion tester. Süddeutsche Zellwolle, A. G., Kelheim Plant, Kelheim. Julian F. Smith. O.P.B. Report PB 3890, 1945; 6 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 387 (Mar. 8, 1946)).

Hollow fiber was made by use of a chemical blowing agent, which gen-

erated bubbles of CO_2 in the fiber. The use of annular spinning nozzle orifices (a round hole with a fine wire in the center) is technically feasible, but the resulting fiber with a continuous lumen is unsatisfactory. Ing. Stoll does not believe that Flockenbast can compete with reasonably priced cotton in a peacetime economy, outside of a limited market for certain specialty goods such as printed tablecloths. Annual plants, no matter how cheap their cellulosic wastes or residues may be, are no more economic as sources of cellulose in Germany than in America. Ing. Stoll has written a book (nearly ready for publication) on the relations of fiber properties to yarn and fabric performance. The English title of the book is "Studies of Controllable Viscose Fiber Properties and of Fiber or Fabric Processing as Factors in Performance Value." Among other items the book covers the combined abrasion and flexing test (a description of which has been published in *Melliand Textilberichte*). Table of contents of the book is included in the report.

Text. Research J. May 1946

Keratin Research

Records of research at the Hochschulinstitut für Textilchemie in Mulhouse and the Textil und Gerbereitechnisches Laboratorium of Karlsruhe Technische Hochschule. Julian F. Smith. O.P.B. Report PB 3889, 1945; 2 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 333 (Mar. 1, 1946)).

Report on interview with Dr. Egon Elöd of the Textil und Gerbereitechnisches Laboratorium. Among the processes developed by Dr. Elöd are an improved non-mercury carroting process for animal hair used in making felt hats; a surface acetylation of viscose rayon; a method for making hollow fiber; and a hydrolysis process for dekeratinizing wool. As an outcome of this search on keratin, Dr. Elöd has developed a process for converting horns and hoofs into an edible white powder with only 50% loss of the initial cysteine content. The report states that the powder is an excellent starting material for making protein medicinals, *e.g.*, silver protein preparations.

Text. Research J. May 1946

Pile Fabrics

Pile fabrics, Johs. Girmes & Co., Oedt, Germany. John J. Repp, Jr. O.P.B. Report PB 3873, 1945; 3 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 387 (Mar. 8, 1946)).

This company, with its subsidiaries, comprises the largest organization of pile-fabric weavers and finishers outside the Russian area in Germany. The main products are: Wollin-imitation wool, transparent velvet, and material simulating animal furs for women's coats. Processes and machinery used are described.

Text. Research J. May 1946

German pile fabrics for upholstery and wearing apparel. John J. Repp, Jr. O.P.B. Report PB 3881, 1945; 10 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 387 (Mar. 8, 1946)).

This report contains descriptions of the following: preparatory processes to weaving, warp preparation, filling preparation, weaving, inspection, mending and measuring, dyeing, and finishing processes. Looms and other machinery used are also described. Diagram of drive for hinge-box-motion is included. Only novel features seen in the German pile-fabric industry were two looms made by the Güsken Loom Works, Dülken, Germany. These were sent to U. S. for further study.

Text. Research J. May 1946

Textile Education

The knitting courses at the Technikum für Textilindustrie Reutlingen (Württemberg), Germany. A. Ward France. O.P.B. Report PB 3885, 1945; 10 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 333 (Mar. 1, 1946)).

A study of the knitting courses at the Technikum für Textilindustrie, the only textile school in Württemberg. Descriptions of the knitting and spinning laboratories with their equipment are given.

Text. Research J. May 1946

Wool-Type Acetate

Records of work on wool-type acetate staple fiber: Drawinella, a Wacker product. Julian F. Smith. O.P.B. Report PB 3887, 1945; 3 pp.; microfilm, 50¢—photostat, $1.00 (through *Bib. Sci. and Ind. Reports* **1**, 333 (Mar. 1, 1946)).

Report of interview with Dr. Schmidt of Alexander Wacker, Ges. für Elektrochemische Industrie, G.m.b.H., Munich, Germany. Drawinella is a wool-type acetate staple fiber, crimped in a soap bath by boiling about 30 min. after cutting dry-spun acetate to the desired staple length. The methods of making Drawinella are covered by patents and are not new. Other Wacker products included polyvinyl alcohol sizing compositions (covered by patents of the Consortium für Elektrochemie), some viscose rayon, a line of vinyl resin plastics, and some Cu salts for pest-control.

Text. Research J. May 1946

MISCELLANEOUS

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Allyl Resins

Allyl chemicals for many new plastics. R. D. Bright. *Pacific Plastics Mag.* **3**, No. 8, 35–6 (1945) (through *Chem. Abstr.* **40**, 1058[4] (Feb. 20, 1946)).

A survey. 11 references.

Text. Research J. May 1946

Broken Picks

Various reasons for broken picks. H. E. Wenrich. *Rayon Text. Mo.* **27**, 28–9 (Jan. 1946).

Many causes of broken picks and suggested remedies are given.

E. G. Martin

Text. Research J. May 1946

Bureau of Polymer Research

The Bureau of Polymer Research. Anon. *Polymer Bull.* **1**, 1–12 (April 1945).

A description of the organization, activities, and research facilities of the Brooklyn Polytechnic Institute's Bureau of High Polymer Research. E. D. Klug

Text. Research J. May 1946

Card Clothing

Card clothing. E. B. Handley. *Text. Weekly* **37**, 234, 236, 238 (Feb. 1, 1946).

The history of the making of card fillet and a brief description of modern methods of manufacture. H. J. Burnham

Text. Research J. May 1946

Coloring Plastics

The coloring of plastics. C. R. M. Oehlcke. *J. Soc. Dyers and Colourists* **61**, 306–10 (1945) (through *Chem. Abstr.* **40**, 1058[5] (Feb. 20, 1946)).

It is shown that the more modern plastics are best colored with insoluble lakes or pigments. Plastics can be colored in 4 ways: (1) dyeing the surface, (2) lacquering of the surface, (3) using soluble dyes, and (4) using organic or inorganic pigments. Method (1) is used only with casein plastics for dyeing buttons with acid, basic, and direct dyes. An interesting adaptation of (1) is in the production of multicolored effects. Methods (3) and (4) are the principal ones for coloring plastics. Features of coloring casein plastics, $PhOH–CH_2O$ plastics, cast resins, cellulose acetate and cellulose nitrate, polyvinyl chloride, polystyrene and polymethacrylate, and polyethylene are noted. Fastness properties should include: fastness to heat, chemical stability to conditions likely to be set up in cure or working operations, and fastness to solvents as well as fastness to light. The poor fastness to light of most of the lakes of acid dyes is tending to limit the choice of dye to the pigment dyestuffs. Demands for yellow, orange, scarlet, red, blue, and green shades are met fairly satisfactorily, though few possess the high-fastness in pale tints. Best results are obtained in brown shades with mixtures, preferably mixtures made during manufacture. Recent patent literature is surveyed. The 3 ways of making colors nonbleeding in solvents are: (1) by introducing acid groups, e.g., sulfonic groups and making a Ba or other metal lake, (2) by introducing acidic, nonlaking groups, e.g., nitro and carboxyl groups, and (3) by increasing the size of the molecule.

Text. Research J. May 1946

Export Trade

Export market review: U. S. A. Anon. *Silk and Rayon* **19**, 1234, 1236, 1238 (Nov. 1945).

A brief résumé of the imports of textiles from the United Kingdom to the United States is given. Tables show, the imports to the United States from the United Kingdom, values in dollars, during 1937–1939 for wool, flax, etc.; cotton and jute manufactures; and the balance of trade between the U. K. and the U. S. A. for 1935–38. A. L. Merrifield

Text. Research J. May 1946

German Textile Developments

Textile developments in Germany. Anon. *Am. Dyestuff Reptr.* **34**, 417–22 (Oct. 22, 1945).

Abstracts are presented of preliminary reports by the textile subcommittee of the Technical Industrial Intelligence Committee on significant technical progress in textiles made by Germans. Some of the developments described are immersion suits, chemical processing of flax fiber and fabric, testing methods, camouflage printing, and dyeing and finishing methods for cotton, wool, and rayon. K. S. Campbell

Text. Research J. May 1946

Mechanical Cotton Picking

Mechanization of cotton production relative to processing for marketing. Francis L. Gerdes. *Textile Bulletin* **69**, 34, 36, 39, 60–1 (Dec. 15, 1945).

A discussion of changes which are said to be in store for the cotton industry as a result of mechanical picking. It is predicted that the flame cultivator will supply the missing link in the chain of mechanical operations incident to complete mechanization of the cotton crop and will increase the demand for mechanical cotton pickers providing cleaner fields at harvest time with a reduction in foreign matter from grass and weeds in the cotton. The importance of providing means for reducing the trash content of machine-picked cotton by the development of especially adapted varieties, induced defoliation of plants prior to harvesting, and development of cleaning equipment at the gin is stressed. There have been 100 spindle-type pickers in operation this past season with a combined capacity of more than 20,000 bales. It is stated that machine-picking preserves yarn strength and appearance as well as hand-picking and that the only detriment is a 5% higher manufacturing waste than corresponding hand-picked cotton. H. J. Burnham

Text. Research J. May. 1946

Mill Lighting

Fluorescent lighting in textile mills. Leonard H. Harrison. *Cotton* **110**, 89, 90, 91 (Jan. 1946).

A description of the replacement of the incandescent lighting system at Smyre Mfg. Co. by a modern fluorescent installation. Calculations are given showing how the job was engineered to give 15 ft.-candles of light in the card and picker rooms and 30 ft.-candles in the rest of the mill. H. J. Burnham

Text. Research J. May 1946

Pirn Winder

New, fully automatic, pirn winding machines. Anon. *Text. Recorder* **62**, 43–5 (Dec. 1945).

Two new pirn winders, one for cotton, woolen, and linen yarns and the other for rayon and silk yarns, are described. The machines incorporate many improved and new features such as: revolving-

type head; spindleless type, suitable for all types of filling packages which are held by centrifugal force; feeler disc independent of thread guide, controlling the diameter through frictional contact; cone former, allowing the winding of pirn with or without cones at bases of pirn; adjustment screws for regulating diameter of package; layer locking device, positive driven, bunch builder mechanism; and pirn ejector for the cotton machine. Machine consists of 10 single, independently controlled units with spindle speeds of 2,800 to 5,000 r.p.m. and a stop motion on each spindle unit in event of yarn breakage. Yarn can be unwound from any type of package. Automatic ejection is not recommended for silk and rayon. A. L. Merrifield

Text. Research J. May 1946

Quartermaster Corps Research

Problems for future Quartermaster textile research. S. J. Kennedy. TEXTILE RESEARCH JOURNAL **15**, 413–22 (Nov. 1945). *Am. Dyestuff Reptr.* **34**, 511–15, 519 (Dec. 3, 1945).

The Research and Development Branch of the Office of The Quartermaster General has during the war carried on a large amount of applied research in the field of textiles —fibers, fabrics and finishes. Accomplishments in a number of categories of great importance to the Army are discussed and particular stress is laid upon fundamental studies suggested by each of the particular problems; future Quartermaster research is projected along these lines. Water-resistance problems have led to an extensive study of the swelling properties of the cotton fiber, as well as of the principles of design of water-resistant fabrics; improvement in field sleeping equipment calls for better insulating material; a complete restudy of webbing construction and materials with the purpose of reducing weight (especially when wet) is in progress; improvement in tentage is expected from research dealing with the action of solar radiation on fabrics, and from studies of tent design and construction; the changes in physical properties of wool and wool fabrics resulting from shrink-resistant treatments are being studied further; a study of the basic factors involved in the failure of clothing seams (one of the least satisfactory elements in military clothing) is planned as a major research project. Other important problems described are abrasion-resistance, tropical deterioration, and antigas and flameproof clothing. Attention is particularly directed to the physical properties of textile fibers and fabrics and the fundamental laws governing their performance under many different conditions. K. S. Campbell

Text. Research J. May 1946

Radio-Frequency Heat

Radio-frequency heat for farm waste plastics. Fritz Rosenthal. *Pacific Plastics* **3**, 38 (Nov. 1945) (through *Bull. Inst. Paper Chem.* **16**, 153 (Dec. 1945)).

The application of radio-frequency heating to farm wastes for plastic materials has not passed the experimental stage; however, the wide potentialities in this field deserve consideration. The most widely distributed waste materials, such as cellulose or lignocellulose, are characterized by a very low thermal conductivity; other waste materials, such as straw, are characterized by high bulk and large air spaces, which further decrease the thermal conductivity, the property on which the conventional contact heating method depends. The application of radio-frequency permits uniform heating by placing the material into an electric field oscillating at high frequency; it does not depend on the thickness of the cross section and the thermal conductivity. The feasibility of the method has been proved in preliminary experiments employing a wallboard composition of 83% cottonseed hulls and 17% phenolic resin, resulting in a uniform cross section of the preformed disc without cleavage, which is in distinct contrast to a check disc molded by contact heat alone. The preheated cottonseed-hull composition can be easily combined with a decorative surface layer of cloth, wood veneer, etc., in one molding operation. This opens up wide possibilities in the home construction of the future.

Text. Research J. May 1946

Rayon and the British War Effort

Rayon went to war: An industry's national service—the early stages. J. Guthrie Oliver. *Silk and Rayon* **19**, 822–3, 896 (Aug. 1945).

The first of a series of articles on rayon's part in the war. This article gives general information on the Cotton Industry Re-organization Bill, voluntary control by the Central Rayon Office and establishment of the utility scheme for fabrics. In June, 1942, rayon yarn was allocated to be used as follows: 50% for utility, 35% for export, and 15% for non-utility. The public with increased earnings would have been glad to purchase materials outside the utility range; however, this range was wide, as there were at least 90 different cloths without taking into account variations produced by dyeing and finishing. Along with the utility scheme was established the Rayon Council which brought together in one body all the rayon interests from the producers to the merchants. This council was temporary and was followed by the British Rayon Federation which allows the industry to express its views with one voice.

A. L. Merrifield

Text. Research J. May 1946

British Rayon Exports

Rayon went to war: The export increase—meeting vital needs abroad. J. Guthrie Oliver. *Silk and Rayon* **19**, 990–1, 994–5 (Sept. 1945).

The second of a series. A brief discussion of the transition of the United Kingdom from "export or die" to strict allocations. During the "cash and carry" period, 1939–41, it was necessary to export in order to pay for materials; however, "lend-lease" changed this, and allocations were arranged so that the urgent needs of the empire were

filled. 7 tables are presented; tables 1–3 show the export of rayon singles yarn from the United Kingdom to the chief destinations, and tables 4–7 are devoted to the export trade in rayon fabrics. Tables 1, 2, and 4–6 are for the 5 years ending in 1944; whereas tables 3 and 7 compare the 5-year periods ending in 1939 and 1944. A. L. Merrifield

Text. Research J. May 1946

World Rayon Production

World rayon production and consumption. J. Guthrie Oliver. *Silk and Rayon* **19**, 924–5 (Sept. 1945).

Review of world import trade in rayon yarns covering South American changes, British Empire trade, and statistics for Asia. A table summarizing the world imports of rayon from 1937 to 1944 is presented and is subdivided into the British Empire, the countries of Europe, Asia, Africa, and of the Americas. A. L. Merrifield

Text. Research J. May 1946

Rotproofing Textiles in India

The rotproofing of heavy textiles. Ordnance Laboratories, Cawnpore, India. *Indian Text. J.* **55**, 721–4 (July 1945) (reprinted from *J. Sci. and Ind. Research*).

In Burma and Assam, untreated heavy textile stores, such as tentage, camouflage nets, sandbags, cordage, portable river-crossing equipment, etc., are rendered unserviceable in a month or so, owing to microbial attack. The nature of fiber damage caused by microbiological agencies is discussed briefly. A chart is produced giving a classified list of the more important rotproofing treatments considered for use in India. Most of these treatments, however, require chemicals that have been in short supply during the war emergency, so the problem has been to conserve such materials as copper and creosote by partial substitution with indigenous materials. Recommendation of any treatment is governed by availability of the chemicals required, ease of application, and general effect of the treatment on the serviceability of the store and on the intrinsic properties of the fiber. Effect of light is a further factor to be considered. A process has been formulated for vegetable dyeing to an approved camouflage shade, using indigenous tannins, the dyes having a certain copper content, with a resultant rotproofing effect that renders the goods very serviceable. Several simple methods for treating large stocks of textiles already fabricated but not rotproofed have been developed for use in operational areas, including application of a plasticized pitch, applied hot; employment of an emulsion of used gear oil, which is always available where there is motor transport; and use of bituminous emulsions which "break" when applied to a fabric surface, to give an impervious coating of bitumen on the fibers. For sizes based on polysaccharides, a mixture of sulfur with a copper salt such as the sulfate or basic carbonate was found useful, although imported salicylanilide is the first choice. When proteins are present (e.g., glue and casein), metallic derivatives and strongly polar compounds are not applicable. Organic compounds, such as the substituted phenols, are most effective, although the constituents of certain indigenous essential oils such as thymol are useful. Methods for evaluating protective effect of treatments include soil burial and tests for resistance to attack by *Chaetomium globosum* and by *Aspergillus fumigatus*. R. K. Worner

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Research Study

Science in war-time England and America. (A talk given at the Rotary Club of Bombay.) Nazir Ahmad. *Indian Textile J.* **55**, 796–802 (Aug. 1945).

As a member of the Indian Scientific Mission, the author visited universities and Government and industrial research laboratories in England and America. The types of research in progress in the two countries, the organization of the laboratories, and the status of scientists are described. Suggestions are given for applying the results of findings to the establishment of similar organizations in India to develop its natural resources and to improve the condition of the people. R. K. Worner

Text. Research J. May 1946

Whole Cotton

The growing and pulping of whole cotton. Frank K. Cameron. *Chemurgic Digest* **4**, 305, 307–13 (Oct. 15, 1945) (through *Bull. Inst. Paper Chem.* **16**, 134 (Dec. 1945)).

Industrially, the growing and processing of whole cotton is a distinctly different enterprise from the growing of cotton for lint. The purpose is the production of a cheap source of cellulose, at the same time conserving the oil from the seeds. To achieve this purpose, planting, cultivating, and harvesting methods have to be modified. The whole cotton plant is reduced in a hammer mill or similar equipment into small fragments, resulting in a fluffy mass from which dark-colored substances may be extracted by organic solvents or aqueous solutions of alkalies; sodium sulfide is very effective. The oil is removed by solvent extraction; all the common fat solvents can be employed. The most satisfactory solvent was a low-boiling petroleum oil. The fibrous residue was pulped by the sulfite, sulfate, and the soda processes; however, if increased pressures were not employed, the pulps contained occasional undigested stem fragments. A 3% nitric acid solution at barometric pressure and 80°C was found to give the most satisfactory laboratory results. Bleaching of the pulp is easily accomplished with sodium hypochlorite. Esters, ethers, xanthates, and dispersions in cuprammonium and alkali solutions have been studied in comparison with like preparations from commercial celluloses, and no essential differences were observed. The present studies seem to indicate that the processing of whole cotton on a commercial scale is practical. 25 references.

Text. Research J. May 1946